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SST Technology Follow-On Program–Phase II

DEVELOPMENT AND EVALUATION OF FUEL TANK SEALANTS

Marlan Pollock

D6-60282
December 15, 1975

FINAL REPORT
Task II

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Prepared for
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Supersonic Transport Office
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16. Abstract

The most promising candidate for sealing fuel tanks of the U.S.A. supersonic transport is a fluorosilicone designated DC 77-028 and DC 94-529. The sealing system was exposed to various environments and the effects evaluated on appearance, physical properties, and sealing effectiveness. Test specimens were exposed to more than 36 000 hr of 219° to 227°C (426° to 441°F) fuel vapor in an accelerated cycle and more than 8000 flight cycles. A sealed tank simulating SST construction was exposed to more than 16 000 hr of 219° to 227°C (426° to 442°F) fuel vapor in an accelerated cycle and ~6500 torsional load cycles each at -46°C (-50°F), +232°C (+450°F), and room temperature. Confidence was developed that the DC 77-028/DC 94-529 fluorosilicone system would perform for 50 000 flight-hours as an integral fuel tank fillet sealant in a Mach 2.7 commercial supersonic airplane. Further development is necessary, however, to achieve this level of confidence for faying surface and injection sealant systems.

about

Progress was also made in developing a backup sealant based on fluorocarbon, and exposing and testing were conducted on a fluorosilicone-fluorocarbon hybrid developed by Dow Corning under an Air Force Materials Laboratory contract.

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PREFACE

Among the agencies participating in this program by furnishing samples for test, doing developmental work, and exchanging information were Dow Corning, General Electric, Products Research and Chemical Corporation, the Air Force Materials Laboratory, and NASA-Ames. The Boeing Company is especially grateful to the Dow Corning Corporation for its continuous interest, diligence, and close cooperation.

The work was administered by the Supersonic Transport Office of the Department of Transportation. The technical monitors were C. R. Ritter and Charles Troha. The work was performed at the Renton Materials Technology Laboratory, the hazardous test cell at Boeing Field, Seattle, and the Materials Technology laboratories at Wichita.

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1.0 INTRODUCTION

A major technological requirement of the U.S.A. supersonic transport program was to ultimately provide a commercial supersonic aircraft which would be competitive from a maintenance standpoint with current subsonic aircraft. The need for an elastomeric material to function as an integral fuel tank sealant in the environment generated by a Mach 2 7 commercial supersonic aircraft was a major requirement which necessitated an extensive sealant research and development program by both The Boeing Company and its material suppliers.

Polysulfide-type sealants now used in subsonic jet aircraft have performed satisfactorily for more than 55 000 hr of flight time on individual aircraft. Fuel tank leakage caused solely by sealant degradation is almost nonexistent and, if mechanical damage to the sealant occurs, repairs are easily accomplished.

Integral fuel tank sealant requirements for the SST impose technical and environmental requirements far beyond the capabilities of the subsonic polysulfide sealants. The SST sealant material must be able to accommodate structural strains over a temperature range of -46° to +232° C (-50° to +450° F) under tank conditions of hot fuel and fuel vapor. It also must adhere to titanium alloy 6Al-4V and be inert to both the titanium alloy and the fluids encountered in manufacturing and flight operations. Finally, it must function satisfactorily for at least 50 000 flight-hours. A search for such a sealant was begun in 1961 through literature search and supplier contacts. Every available sealing system in use on supersonic airplanes was investigated to ascertain its suitability. When the SST fuel tank sealing program was initiated in 1965, it was obvious that only two elastomeric polymer systems could reasonably be considered as leading candidates on the basis of thermal stability and resistance to fuels. These were the fluorocarbons and the fluorosilicones.

Even though their thermal and fuel resistances are excellent, the fluorocarbons such as the Fluorels and Vitons have a tendency to cause stress corrosion of titanium and currently depend on being suspended in solution for proper application. Volatilization of the solvent precludes their effectiveness as a filleting sealant, since they must be applied in thin layers to prevent excessive porosity from outgassing. The fluorocarbons also have unacceptable low-temperature flexibility.

On the other hand, at low temperatures the fluorosilicones exhibit excellent properties which get progressively poorer as the temperature increases. Under conditions of high temperature and pressure, they tend to revert to a gummy mass. In 1967, Dow Corning introduced a fluorosilicone which was a significant improvement over the others. Originally, it was designated DC 94-516 which later was changed to DC 77-028. Today it is marketed as DC 94-529.

Dow Corning and Boeing worked closely together to develop the fluorosilicone system with the emphasis on improving reliability of adhesion and repairability and creating satisfactory faying surface and injection sealants. Primary sealing of the SST was to be by means of fillet seals, but in many situations it was necessary to use fillet sealing in conjunction with other sealing methods. The faying surface sealant passed laboratory tests but was suspect because of visible channels through it and failure to seal the cover of a test tank. The problem with injection sealant was one of excessive thermal expansion, which caused it to extrude and to tear itself and the fillet seal covering it.

The basic fillet sealant, designated DC 77-028, was varied to obtain properties that would make it more suitable for faying surface and injection or prepack sealing by using different fillers and combinations of fillers. The faying surface sealant was designated DC 77-053, and the injection or prepack sealant was designated DC 77-066. Figure 1 shows how these sealants are used.

Another obstacle to determination of the suitability of a fuel tank sealant material for SST application was the lack of complete physical property requirements. On this basis, Boeing established the following fillet sealant target for 50 000-hr flight service life in the U.S.A. SST.

Technical Requirements

1.	Adhesion to 6Al 4V titanium (peel and tear resistance)	1.07 kg/cm (6 lb/in.), 85% cohesive failure, minimum
2.	Tensile elongation	15% minimum
3.	Shore A hardness	Less than 20 points change
4.	Weight change	Less than 20%
5.	Volume change	Less than 10% loss
6.	Deflection simulation [0.028-cm (0.011-in.) deflection]	Less than 30% tear after 100 cycles
7.	Leakage (picture frame)	No leaks

Application Requirements

1.	Flow (slump)	1.25 cm (0.5 in. maximum in 2 hr
2.	Extrusion rate	15 g/min. minimum
3.	Cure	Less than 71° C (160° F)
4.	Working life	At least 0.5 hr
5.	Repairability (peel test)	1.07 kg/cm (6 lb/in.), 85% cohesive failure, minimum

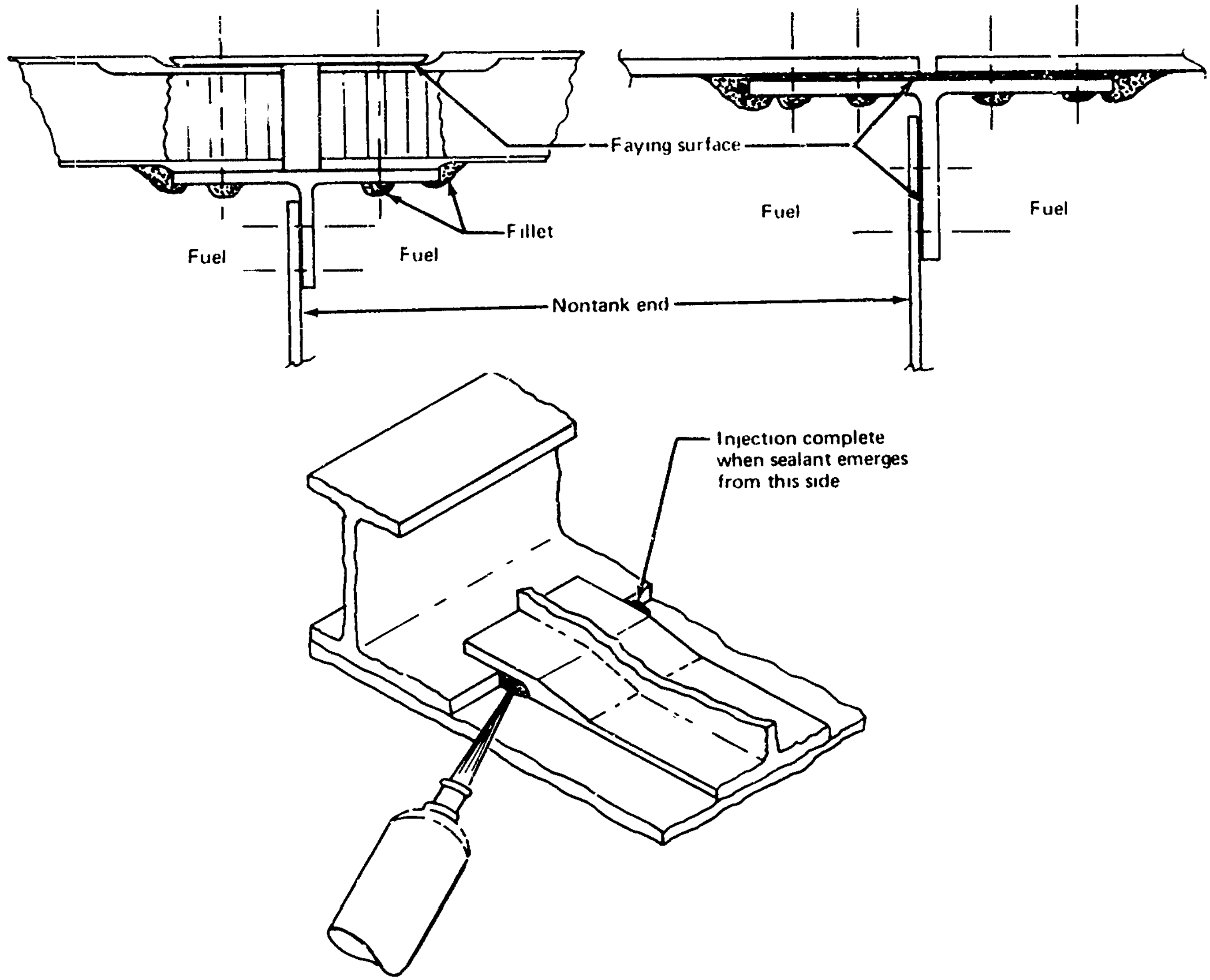


Figure 1.—Typical Fillet, Faying Surface, and Injection Sealing

Work on the basic DC 77-028 fluorosilicone system was conducted through the SST prototype program and through a Phase I follow-on development program, under contract DOT-FA-SS-71-12, awarded by DOT after termination of the SST activities. The results of these efforts led to improvements in the sealant characteristics and test and evaluation criteria as reported in D6-60221, *Development and Evaluation of Fuel Tank Sealants*. Based on these findings, a Phase II technology follow-on program under contract DOT-FA-72WA-2893 was awarded to continue development of the system and demonstrate its capabilities for commercial supersonic aircraft applications. The primary objectives of the Phase II effort were:

1. Conduct 30 000 hr of simulated service testing at elevated temperatures.

2. Verify suitability for 50 000 flight-hours.

3. Develop improved adhesion.

4. Develop repair techniques.

5. Develop faying surface/injection sealant capabilities.

Also as part of the Phase II program, a search was conducted for a backup to the fluorosilicone system. A contract was issued to Products Research and Chemical Corporation to develop a practicable sealant based on a fluorocarbon system pioneered by the Air Force Materials Laboratory. Both NASA and AFML were active in funding fuel tank sealant development programs, and these programs were followed closely by Boeing. Another backup candidate was a hybrid fluorocarbon-fluorosilicone developed by Dow Corning under an AFML contract. AFML supplied samples for test.

2.0 TEST METHODS AND PROCEDURES

The approach was to subject test specimens to various static and dynamic exposures and to measure changes in physical and mechanical properties. Responses of the sealant to the exposures were used as indications of useful life and deficiencies of the basic material or system. Functional tests, which measured sealing ability depending on all properties of the total system, were also conducted to evaluate the practical aspects of the sealant's use.

2.1 CYCLING ENVIRONMENTAL EXPOSURES

Two principal cycling environments were used: flight and accelerated. The test tanks were exposed to cycles similar to the accelerated cycle.

2.1.1 FLIGHT CYCLE

The simulated flight cycle (fig. 2) provides about 50 hr per week of elevated-temperature fuel vapor exposure. The equipment is shown schematically in figure 3 and photographically in figure 4. The loaded specimen rack is shown in figure 5.

2.1.2 ACCELERATED CYCLE

The accelerated cycle (fig. 6) exposes the specimens to a minimum of 140 hr per week of elevated-temperature fuel vapor. The exposure chamber is shown in figure 7 with the test specimens in place.

2.1.3 TEST TANK CYCLES

Environmental exposure cycling of the tank was the same as for the accelerated cycle except that nitrogen at atmospheric pressure was substituted for air at 6.9 kPa (1 psia). The tank was not designed to withstand the near-vacuum pressure.

2.2 PREPARATION OF TEST SPECIMENS

2.2.1 CLEANING OF TEST PANELS

All test panels and parts were cleaned by applying cleaner from clean polyethylene (or equivalent) squeeze bottles. The cleaner consisted of:

	Volume percent
Aromatic naphtha (TT-N-97, type I, grade B)	50
Ethyl acetate (TT-E-751) or isopropyl acetate (TT-I-721)	20

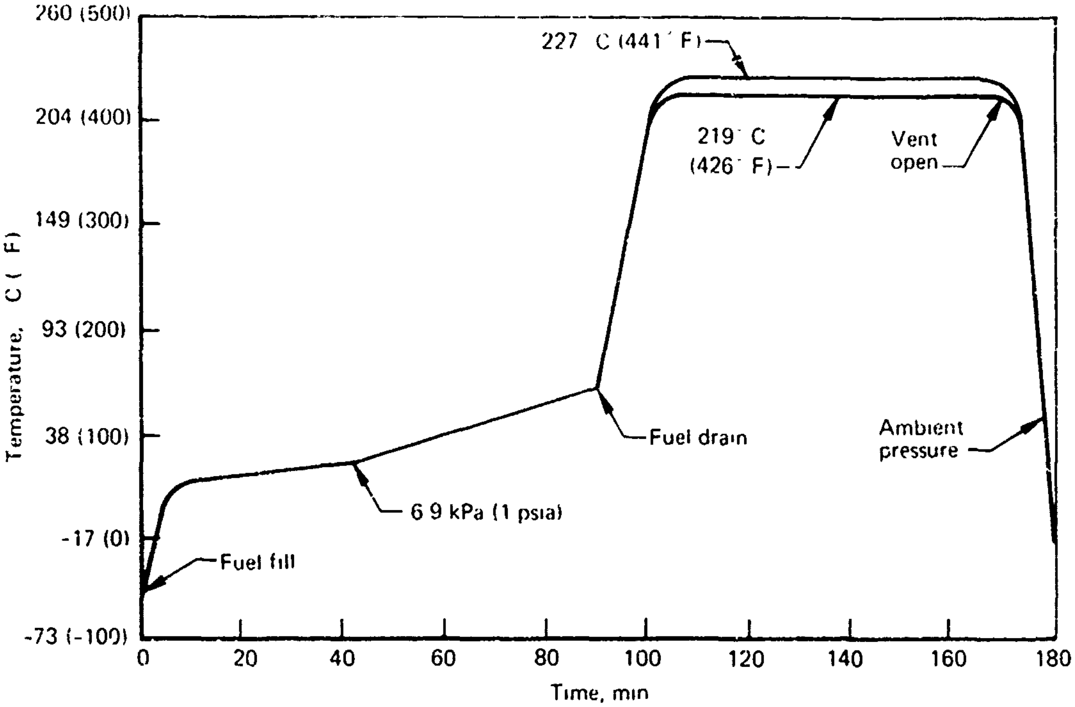


Figure 2.–Standard Flight Cycle

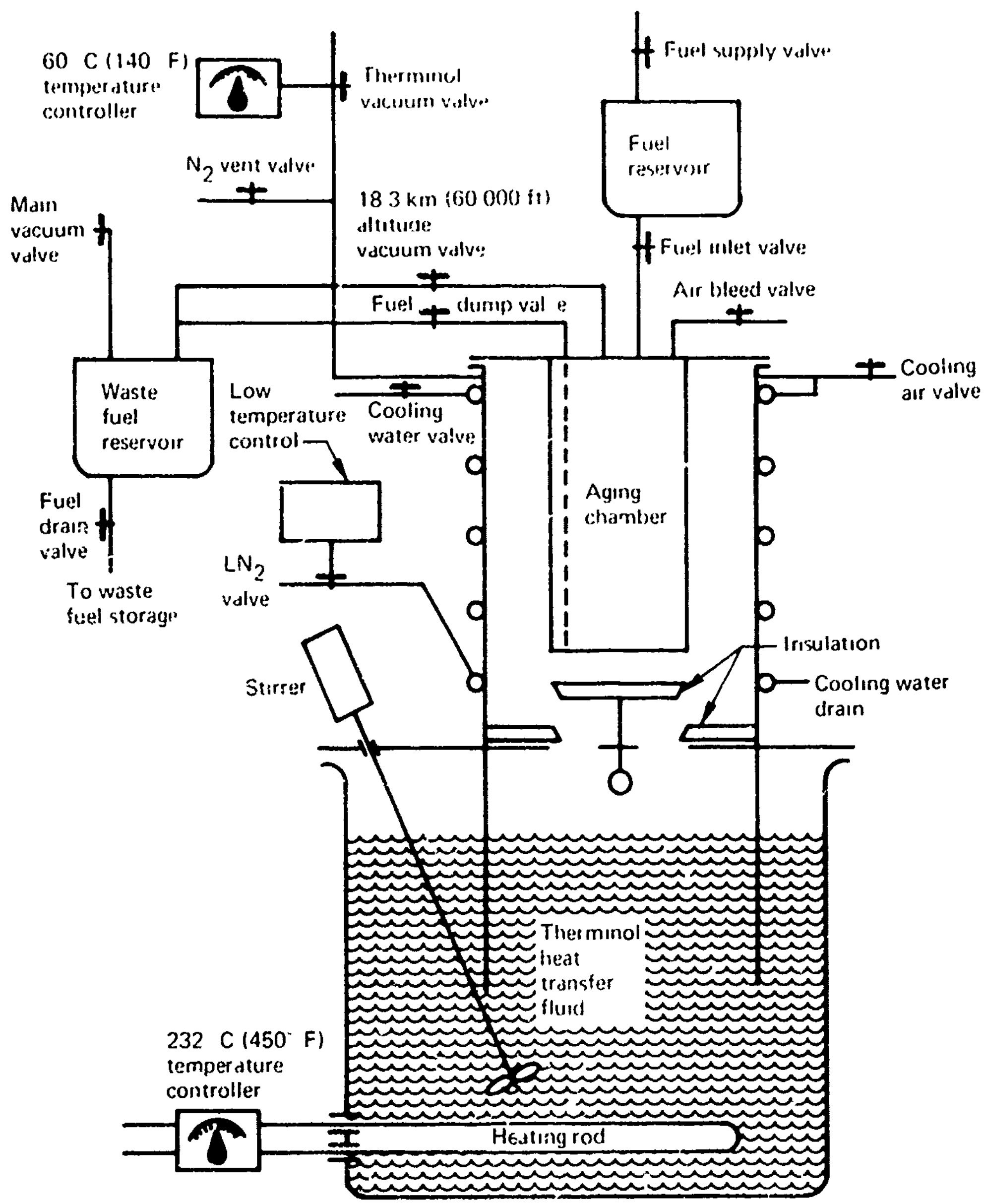


Figure 3.—Flight Cycle Apparatus

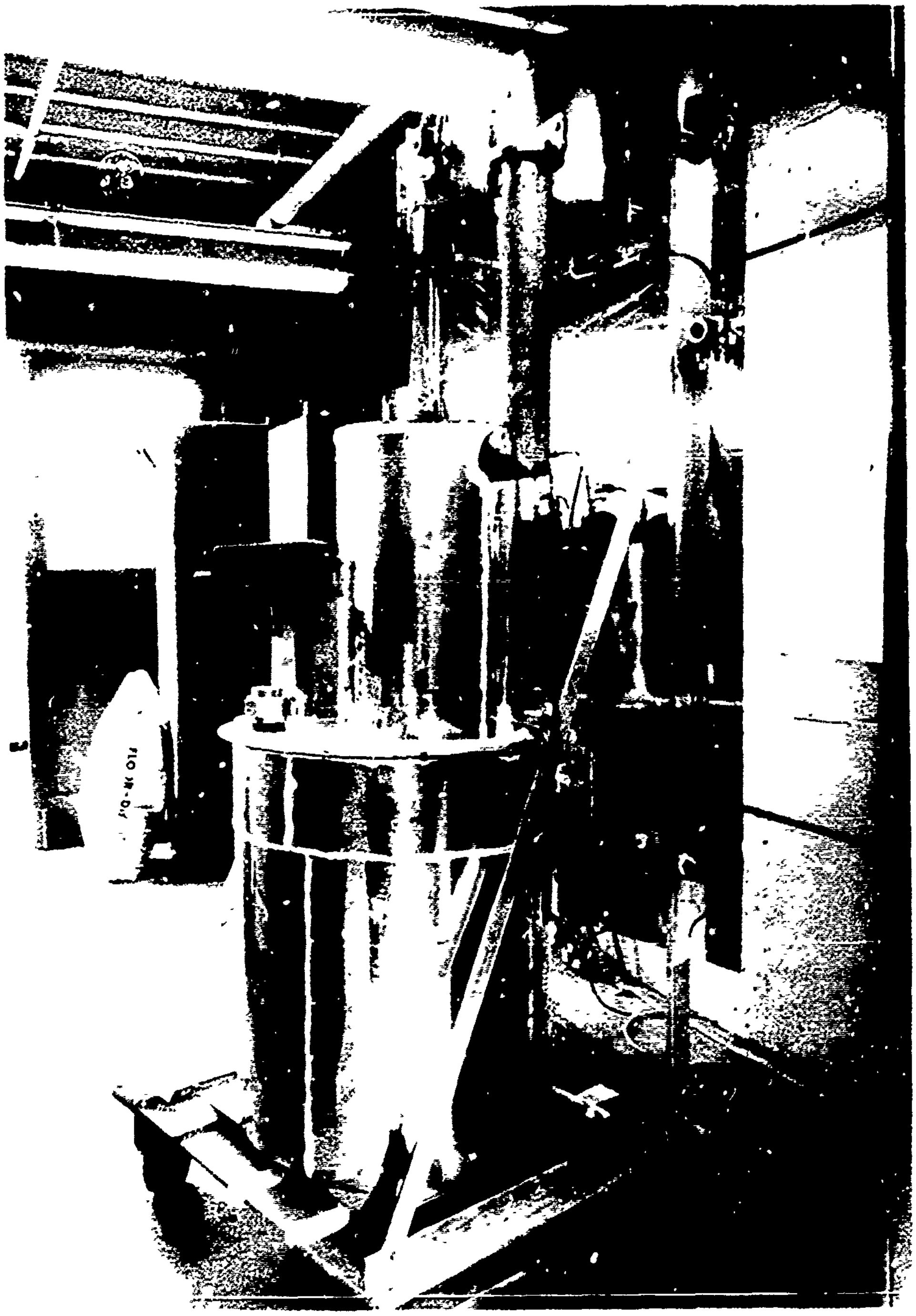


Figure 4   Flight Cycle Apparatus Without Insulation

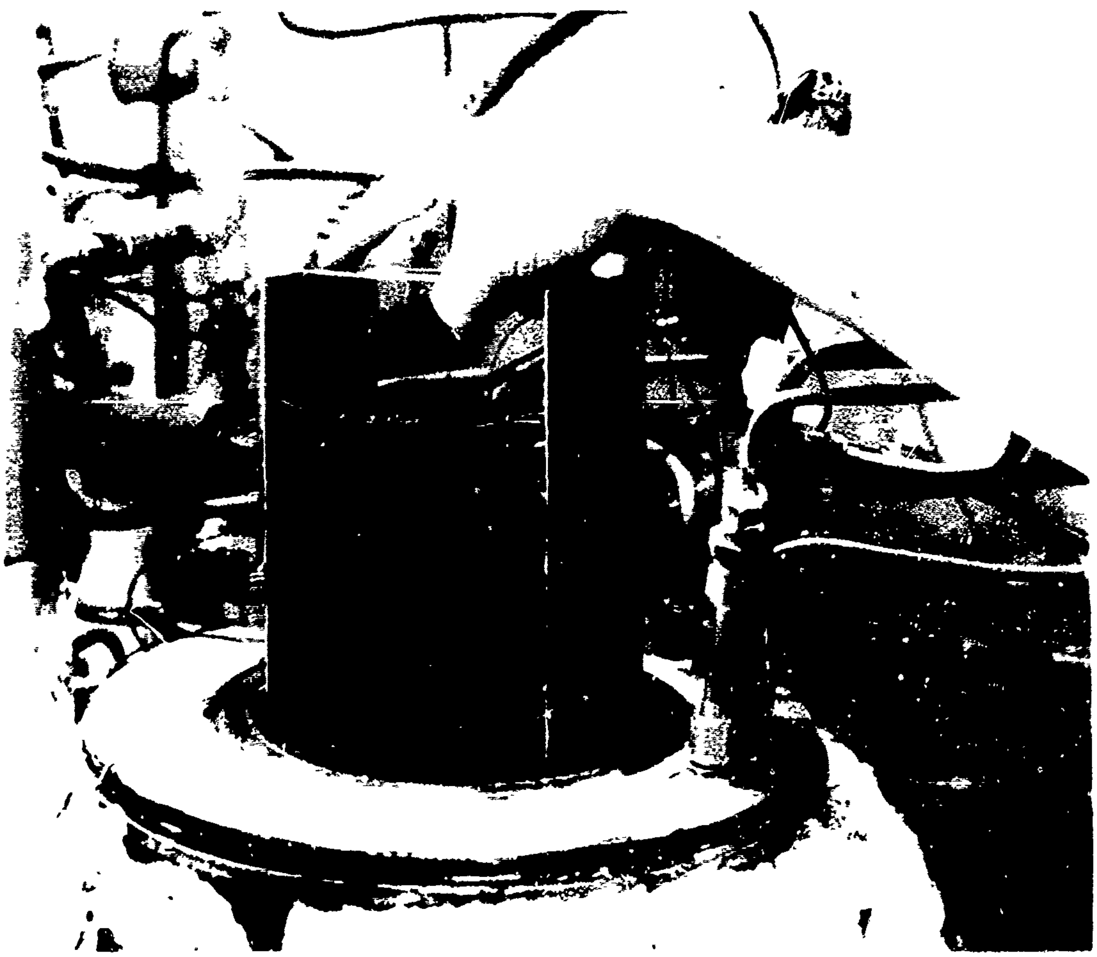


Figure 5. Specimen Loading of Flight Cycle Chamber

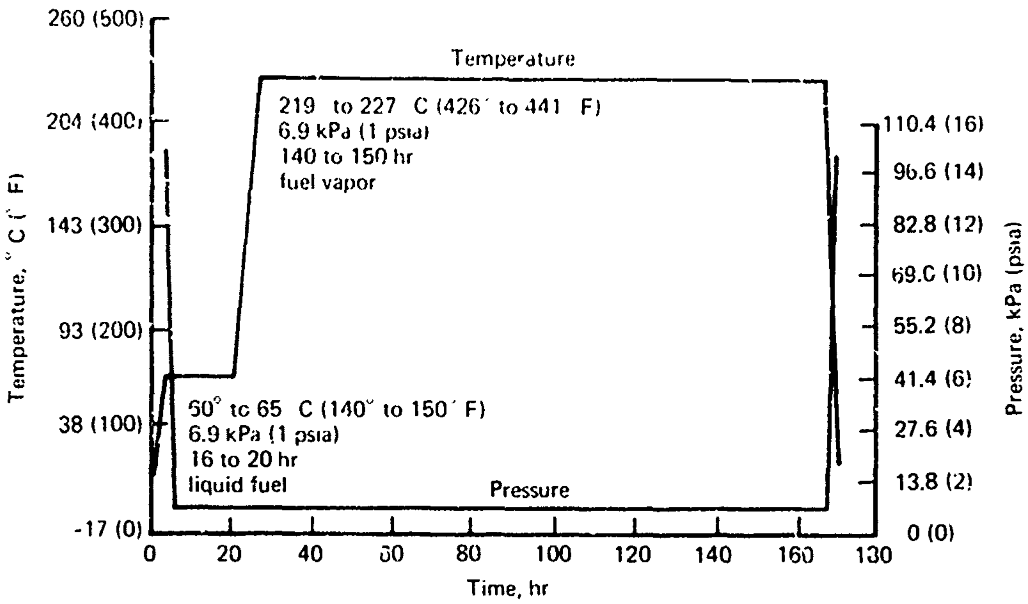


Figure 6.—Accelerated Exposure Cycle

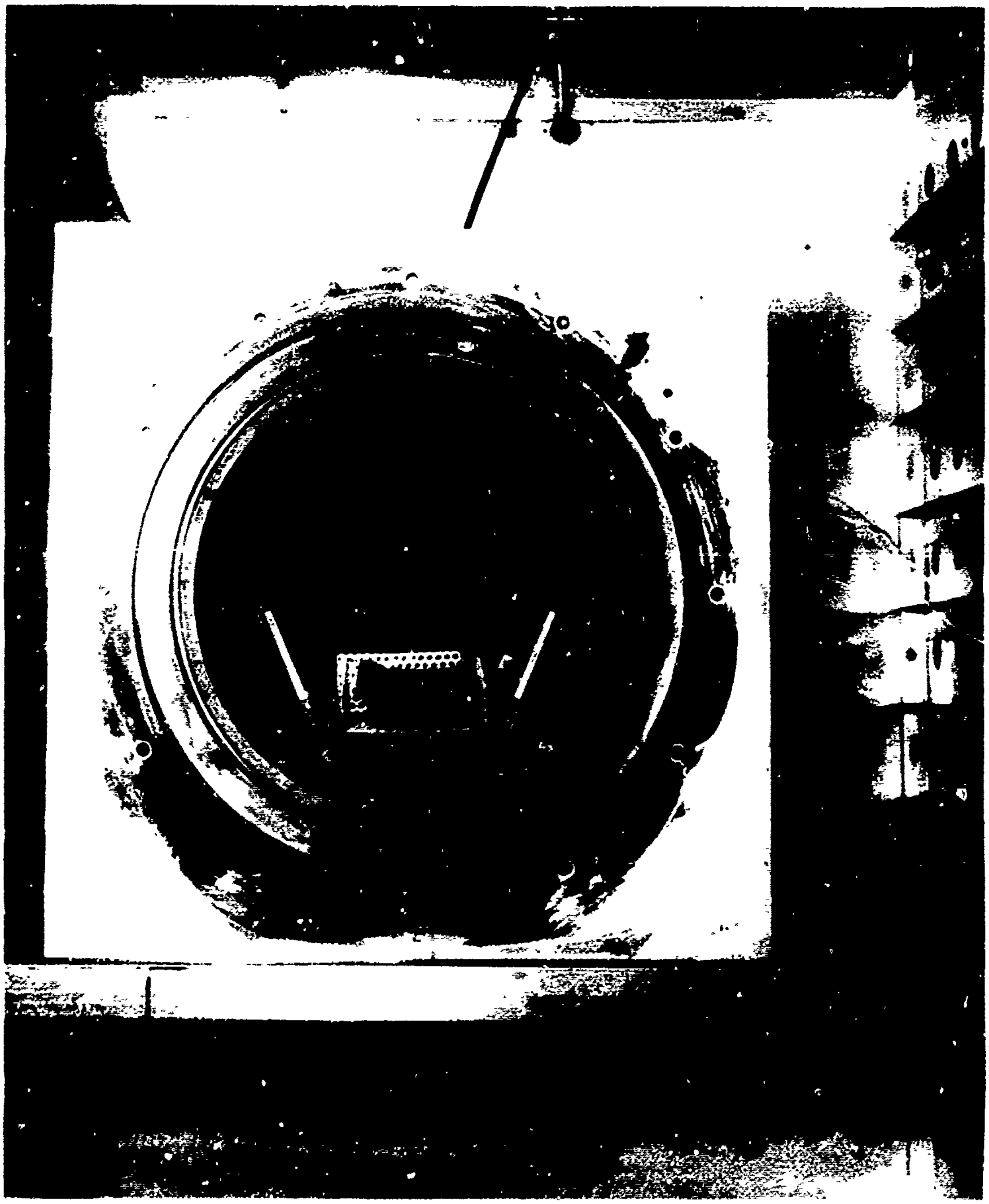


Figure 7.—Accelerated Cycle Exposure Chamber

Methyl ethyl ketone (TT-M-261)	20
Isopropyl alcohol (MIL-F-5566)	10

The cleaner was applied directly from the bottle to the panel or part so that the entire surface was wetted. The surfaces to be cleaned were thoroughly scrubbed with clean gauze pads wetted with cleaner. The solvent was wiped off while wet with clean, dry gauze pads. This procedure was repeated as required to produce a clean surface as determined by no visibly detectable residue on the gauze. Each gauze pad was used for one scrubbing or drying application only. Cleaned panels were not stacked, but were covered with paper toweling or equivalent until used. Test panels and parts were used within 8 hr after cleaning.

2.2.2 APPLICATION OF PRIMER

A coat of primer was applied to cleaned test panels before applying the sealant. The primer was applied in a continuous coat as uniformly as possible using a gauze pad. The primed panels were dried for 30 ± 10 min at 24° ± 1.5° C (75° ± 5° F) and 50% ± 5% relative humidity. Then the panels were immediately placed in an oven and the primer was cured at 232° C (450° F) for 25 ± 5 min. After curing, the primed surfaces were protected from contamination until sealant application. If sealant was not applied within 90 min after the primer was cured, the panels were cleaned and reprimed.

2.2.3 PREPARATION OF SEALANT

Prior to mixing, two-component sealant compounds were stored at 24° ± 1.5° C (75° ± 5° F) for a sufficient time to allow the material to reach a state of equilibrium with that temperature. The activator was added immediately before weighing. Mixing instructions were as follows:

1. Weigh the correct amounts of base and activator onto a clean, flat stainless steel plate or pan immediately prior to mixing. The activator must not be allowed to contact the plate.

2. Handmix the sealant compound by folding and squeezing with a spatula for a minimum of 5 min until the sealant compound appears uniform.

3. Spread the sealant compound on a clean, flat stainless steel plate or pan so that the maximum depth is less than 1.21 cm (0.5 in.). Vacuum degas the sealant compound for 10 min at 1.73 kPa (0.25 psia) or less.

4. Remove the plunger and plug the nozzle end of a cartridge for the Semco 250 gun. Scoop up the sealant with a spatula, place in the open end of the cartridge, and drive down by sharply rapping the nozzle end of the cartridge on something solid. Repeat until the cartridge is filled.

5. Vacuum degas the filled cartridge for 5 min at 1.73 kPa (0.25 psia) or less. Use a plastic film as necessary as an extension of the cartridge to prevent overflow of the sealant. Place the plunger in the cartridge using care to minimize air entrapment.

When required, the sealant was put into refrigerated storage at or below -40° C (-40° F) immediately after being placed into the cartridges. Dry ice was not allowed to be used for refrigeration The sealant was stored at or below -40° C (-40° F) for a minimum of 16 hr but not longer than 72 hr and conditioned at 4.4° ± 1.5° C (40° ± 5° F) for 4 ± 2 hr immediately prior to thawing. It was thawed by vertically immersing the frozen cartridges in a 49° ± 0.6° C (120° ± 2° F) water bath for 4 min ± 5 sec with the plugs installed and the upper end of the cartridge 2.54 cm (1 in.) above the liquid level.

2.2.4 SEALANT CURE

2.2.4.1 Fluorosilicone

Standard cure was 14 consecutive days at 24° ± 1.5° C (75° ± 5° F) and 50% ± 5% relative humidity. An accelerated cure consisting of 24 hr (minimum) at 71° ± 3° C (160° ± 10° F) followed by 1 hr (minimum) at 149° ± 3° C (300° ± 10° F) was sometimes used instead of the standard cure.

2.2.4.2 Fluorosilicone-Fluorocarbon Hybrid

The FCS-210, also known as DC 77-108, was cured 1 hr at 100° C (212° F) followed by 1 hr at 150° C (302° F).

2.2.5 PREPARATION OF SEALANT SLABS

The sealant was cast to a thickness of 0.32 ± 0.02 cm (0.125 ± 0.008 in.) in a closed mold lined with Teflon. The mold was filled by extruding the sealant from a sealant gun with a Semco 440 nozzle. The nozzle was freed of air by a preliminary extrusion of 5 to 7 cm (2 to 3 in.) of sealant. During the casting operation, the tip of the nozzle was placed in an injection mold and was not removed until the mold was filled to excess.

When the standard cure was used, the sealant was kept in the closed mold until the cure was completed or removed at any time after 96 hr. When the accelerated cure was used, the sealant was allowed to remain in the mold at 24° ± 1.5° C (75° ± 5° F) and 50% ± 5% relative humidity for a minimum of 48 hr prior to the 71° C (160° F) exposure. The slabs were removed from the mold before completing the cure at 149° C (300° F).

2.3 TEST PROCEDURES

2.3.1 PHYSICAL AND MECHANICAL PROPERTIES

2.3.1.1 Tensile Strength and Elongation

Ultimate tensile strength and elongation were determined in accordance with ASTM D-412 using a jaw separation rate of 51 cm (20 in.)/min. Miniature specimens were used cut from slabs prepared as described in section 2.2.5. Five specimens were tested for each point except for those from lot 401117 where eight were tested per point.

2.3.1.2 Hardness

Shore A durometer hardness was determined in accordance with ASTM D-2240 taking the median value as the hardness for each specimen. The hardness is reported as the average of four specimens. Volume change specimens were used for hardness measurements.

2.3.1.3 Weight Loss

Specimens approximately 2.5 by 5 cm (1 by 2 in.) were cut from a slab of sealant prepared in accordance with section 2.2.5. Before and after the applicable environmental exposure period, the test specimens were conditioned for 24 hr in a dessicator and then weighed immediately.

Percentage weight loss was calculated as follows:

$$\frac{(W_1 - W_2) \times 100}{W_1}$$

where

W_1 = weight of sample before aging

W_2 = weight of sample after aging

Percentage weight loss for each determination is the average of four specimens.

2.3.1.4 Volume Change

Specimens approximately 2.5 by 5 cm (1 by 2 in.) were cut from a slab of sealant prepared in accordance with section 2.2.5. The volume change of environmentally aged specimens was determined in accordance with ASTM D-471. Volume change is reported as the average of four specimens.

2.3.1.5 Adhesion to Titanium (Tee Peel)

Details of Specimen Preparation.–The required number of 0.127- by 7.4- by 15.2-cm (0.05- by 2.9- by 6-in.) panels were prepared from annealed 6Al-4V titanium alloy. An equal number of 7.4- by 30.5-cm (2.9- by 12-in.) strips were prepared from 200-mesh stainless steel screen.

The panel surfaces and screen were cleaned as described in section 2.2.1 and primer was applied as in section 2.2.2.

The sealant was applied to approximately 12.7 cm (5 in.) at one end of the panel to a depth of 0.318 ± 0.06 cm (0.125 ± 0.025 in.) and leveled, using a suitable jig. The screen was impregnated with sealant for approximately 12.7 cm (5 in.) on one end. The sealant-impregnated end of the screen was placed on the panel so that the loose, unimpregnated end faced the end of the panel free from sealant. The screen was smoothed down on the layer of sealant, taking care not to trap air under the screen. An additional 0.318 ± 0.06 cm (0.125 ± 0.025 in.) coating of sealant compound was applied over the impregnated screen. The sealant was cured in accordance with section 2.2.4.

Testing of Specimen.–After exposure, two 2.5-cm (1-in.) wide strips were prepared on each panel by cutting completely through the screen and sealant to the metal lengthwise along the panel and continuing completely along the unimpregnated screen. The loose end of each 2.5-cm (1-in.) wide strip in turn was clamped in one jaw of a suitable recording tensile testing machine, and the adjacent end of the panel was fastened in the other jaw as shown in figure 8. Cuts through the sealant under the screen were made so that an initial separation of sealant from the metal panel was promoted. The screen was pulled at an angle of 180° from the panel and at a jaw separation rate of 5.1 cm/min (2 in./min).

Cuts in the sealant to the metal panel at the junction of separation were made at an angle of 45° toward the direction of separation at approximately 1.5-cm (0.6-in.) increments (approximately every 24 sec) on the left side of the panel as shown in figure 8. No cuts were required for 100% adhesive failure; however, any cohesive failures were treated as described. On the right side, except for the initial cut to promote separation, cuts were made only as necessary to prevent the sealant from peeling from the screen. All cuts extended completely across the strip being peeled and penetrated completely through the sealant to the panel.

The percentage of cohesive separation was determined from the ratio of cohesive separation area to total cohesive and adhesive separation on both test areas. The cohesive strength was determined during cohesive tear. The average of the cohesive strength, as determined from an extensometer graph of the right-side pull, was recorded. Values recorded during cutting, or while load was being picked up after cutting, were not included in the average. Panels that were environmentally exposed were tested within 24 hr after removal from the exposure condition.

2.3.2 FUNCTIONAL TESTING

2.3.2.1 Picture Frame Shear

Details of Specimen Preparation.–The test panel was cleaned and primed as described. For a faying surface sealed panel, a layer of sealant was applied to one of the mating surfaces and the panel assembled while the sealant was still wet so that a continuous bead was extruded. The extruded sealant was then removed. Sealant was applied to fillet-sealed panels after assembly. A continuous fillet was applied around the periphery of the joint. Curing was in accordance with section 2.2.4.

Testing of Specimens–Leak testing was accomplished by placing a plexiglass box over the T-side of the panel (fig. 9), filling the box with water, and pulling a 13.8-kPa (2.0-psig) vacuum. Leakage was detected by observing the formation of bubbles. The test panel was then assembled in a picture frame fixture for application of structural loads. The frame was placed in an insulated conditioning chamber and loaded from the

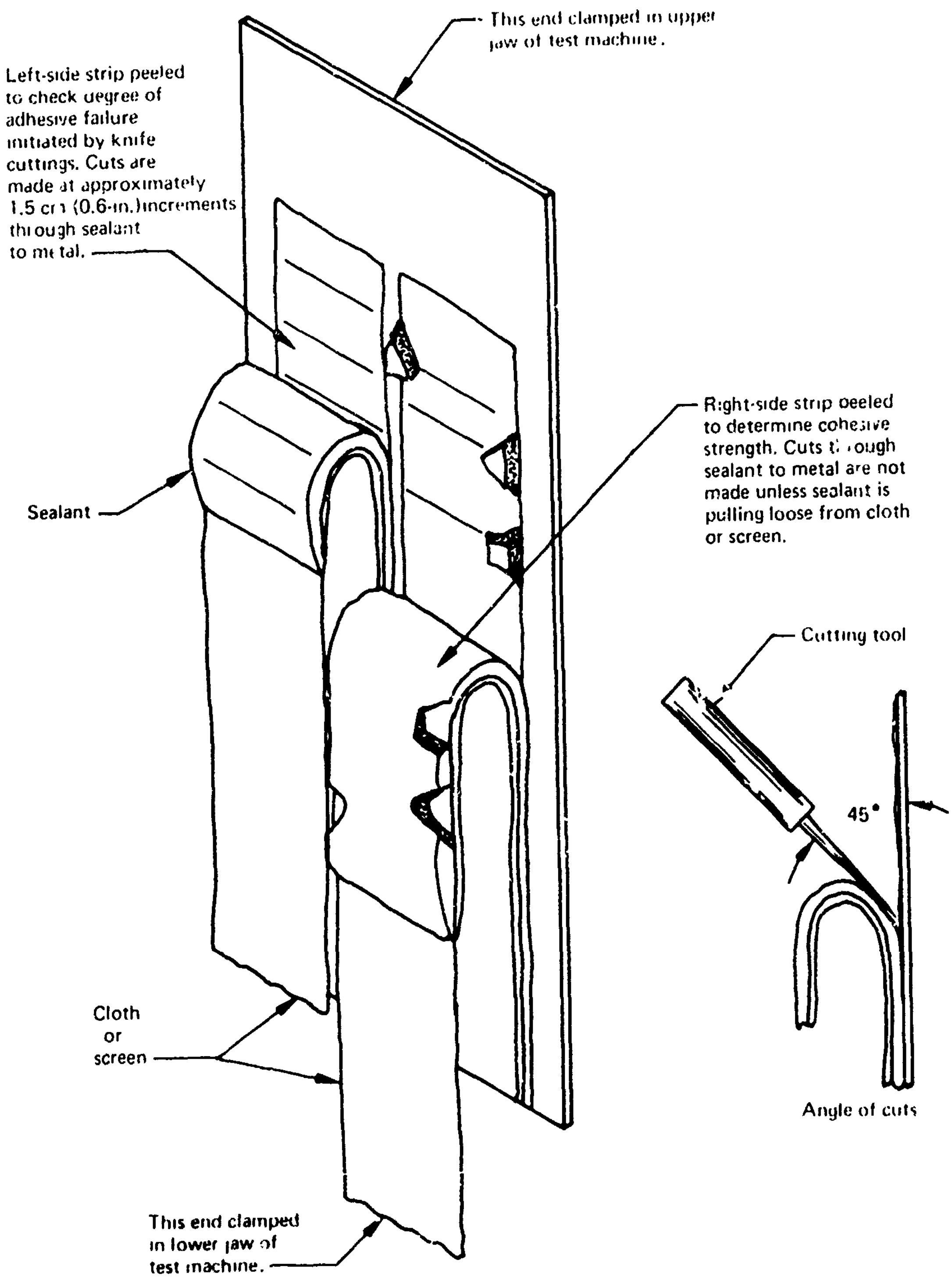


Figure 8.—Peel Strength Testing

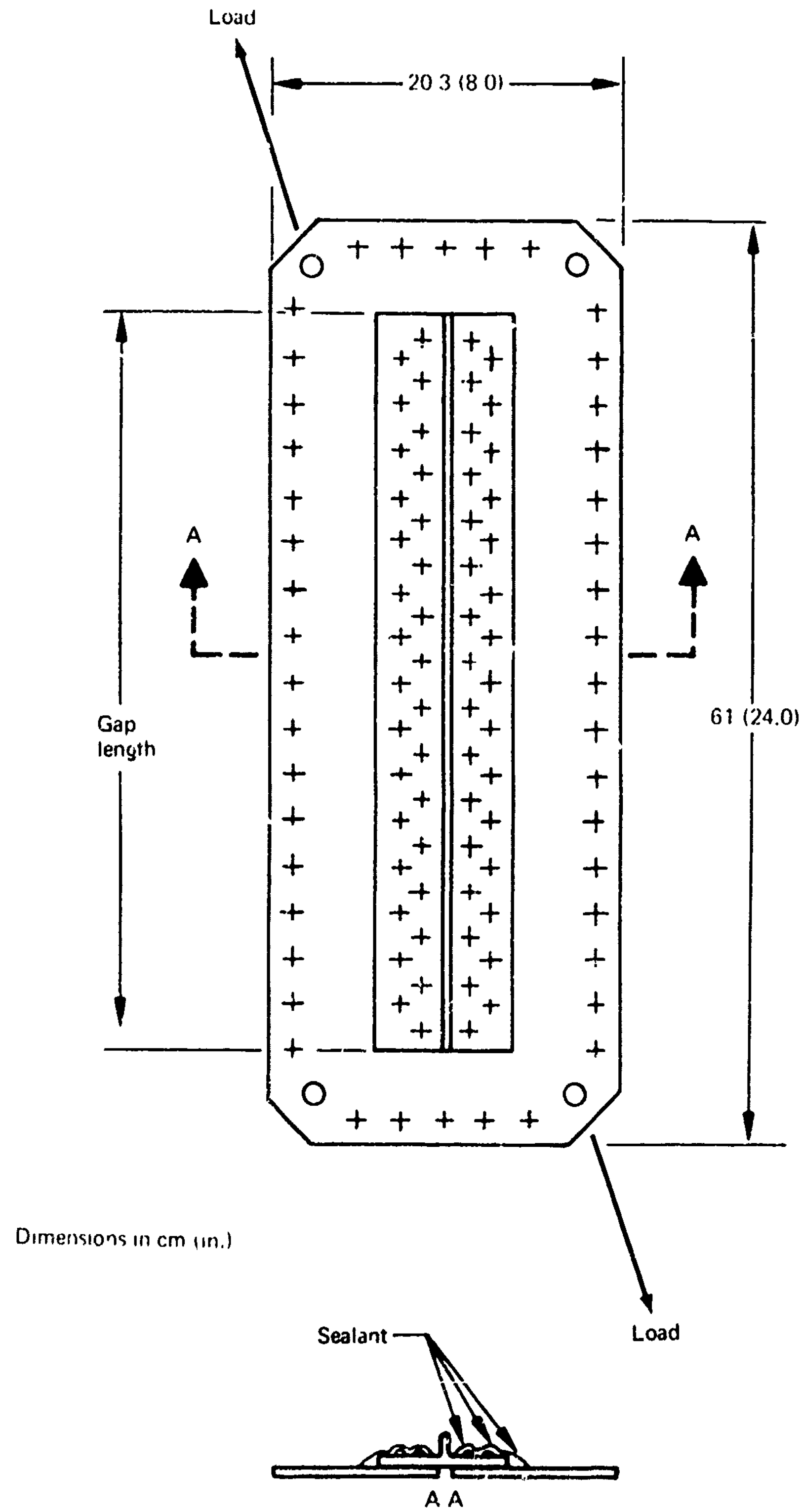


Figure 9.–Picture Frame Shear Specimen

corners. The test apparatus is shown in figure 10. A 22 680-kg (50 000-lb) tension load was applied to the frame 10 times at -46° C (-50° F), 10 times at room temperature, and 10 times at 232° C (450° F). A hydraulic cylinder was used for load application. The 22 680-kg (50 000-lb) load represents 100% of design limit load for the test panel.

After loading, the test panel was tested for leaks as described. The panel was then subjected to environmental aging. After various periods of aging, the panel was subjected to the loading and leak-testing conditions described.

2.3.2.2 Flight Cycling Fillet Deflections

Specimens shown assembled in figure 11 were prepared from annealed 6Al-4V titanium alloy. They were cleaned and primed prior to fillet application according to sections 2.2.1 and 2.2.2. The titanium strips, B, were of constant length. Strips A overlapped by B were varied in length so that the gaps at the juncture of the A strips and B strips would vary with a constant end deflection of the strips B. Deflections were 0.008 cm (0.003 in.), 0.013 cm (0.005 in.), 0.020 cm (0.008 in.), 0.038 cm (0.015 in.), 0.051 cm (0.02 in.), and 0.076 cm (0.03 in.). The six specimens were mounted in a vertical row and fitted to the interior of the lid of the flight cycle apparatus. The end of each of the B strips was deflected at a rate of approximately 2 cycles per minute by means of a rod extending through the lid. The length of tear in each fillet throat was measured each time the flight cycle chamber was opened.

2.3.3 TANK TEST

The tank test was performed by the Wichita Division of The Boeing Company. A complete description of tanks, facilities, tests, and results is given in appendix A.

Figure 10 Picture Frame Test Fixture

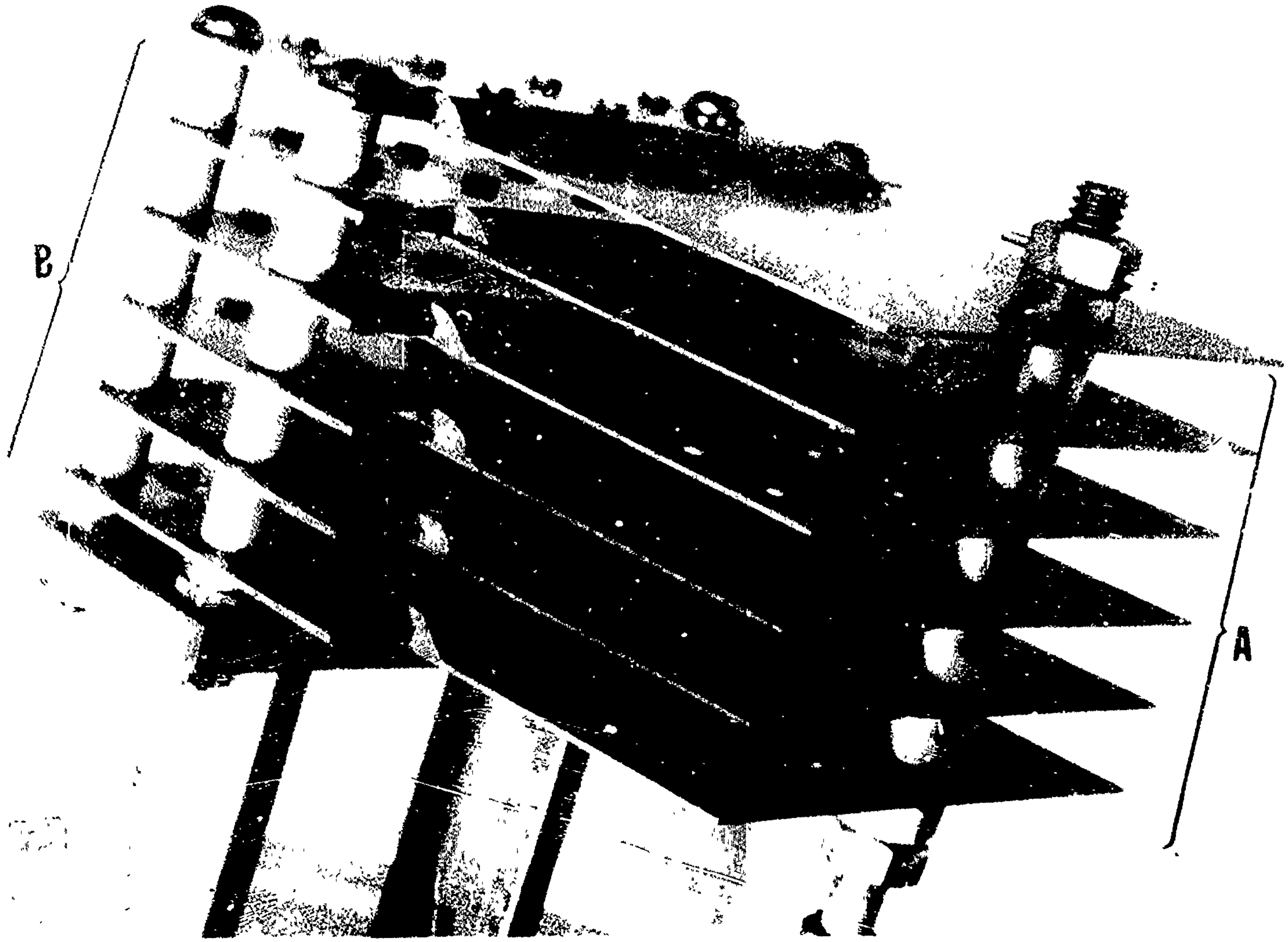


Figure 11. Flight Cycle Deflection Specimen

3.0 RESULTS

Four different lots of DC 77-028 were tested. Only one lot, 401117, was tested in both accelerated cycling and flight cycling. Specimens from lot 1222 were also in accelerated cycling. Specimens from lot 205117 were in flight cycling. The tank used material from lot 205180. All lots were manufactured at different times; the oldest was lot 1222 and the newest was lot 205180. Lot 401117 was a 226.79-kg (500-lb) production lot manufactured to demonstrate production capability and proof of ability to scale-up from laboratory size lots.

The only experimental sealant providing test results was the FCS-210 developed by Dow Corning with AFML funding.

3.1 EFFECTS OF ACCELERATED AND FLIGHT CYCLING

Figures 12 through 26 summarize changes in tensile strength, elongation, volume, weight, and adhesion after various periods of exposure to accelerated and flight cycling. Note that hours at the most severe condition, fuel vapor at 219° to 227° C (426° to 441° F), were used as a common base for comparison. For accelerated cycling, a factor of 1.2 may be used to calculate total exposure time to all fuel tank conditions. For flight cycling, a factor of 3 may be used. Each hour plotted is equivalent to one flight; therefore, 8217 flights were simulated. The plots also indicate the scatter in tensile and elongation data. Considering the inherent scatter, the difference between lots of material appears to be insignificant.

The sealant remained flexible and all but elongation at 232° C (450° F) and volume loss met target requirements after completing the exposures indicated. Elongation and volume loss were very close to the target requirements, and failure to meet them should not be construed as evidence that the sealant would not be serviceable.

The lack of adhesion (peel test) data was due to invalid results, mainly adhesive failure between the sealant and embedded screen rather than between the sealant and titanium substrate.

It was hoped that a correlation of test results from accelerated cycling and flight cycling would be possible so that service life might be predictable using the shorter calendar time accelerated cycle. The indication is, however, that there is not a direct correlation and flight cycling is more severe than accelerated cycling. The comparison of weight and volume loss data in figure 27 dramatically depicts this.

3.2 FUNCTIONAL TESTING

3.2.1 PICTURE FRAME

The condition of the fillet-sealed picture frame test specimen is shown in figure 28 after 28 634 hr of 219° to 227° C (426° to 441° F) fuel vapor exposure in the accelerated cycle and 100 load cycles at each temperature. The sealant was flexible and the panel

Hours of exposure to 219° to 227° C (426° to 441° F) fuel vapor	0	149	287	433	1043	1344	1563	1994	2533	3437	4983	7675	9036	11 427	15 359	20 454	36 856
Shore A hardness	33	46	42	45	40	42	40	32	39	32	38	39	39	40	40	45	56
Peel, lb per in./% coh											95/60						
Weight loss, %		1.1	1.1	1.5	1.9	2.1	0.9	1.8	2.6	3.1	4.4	6.2	6.1	7.5	8.3	10.6	18.6
Volume loss, %		1.5	1.8	1.6	1.5	2.5	1.0	2.1	3.0	3.7	4.9	7.2	6.8	8.4	9.5	10.7	20 0

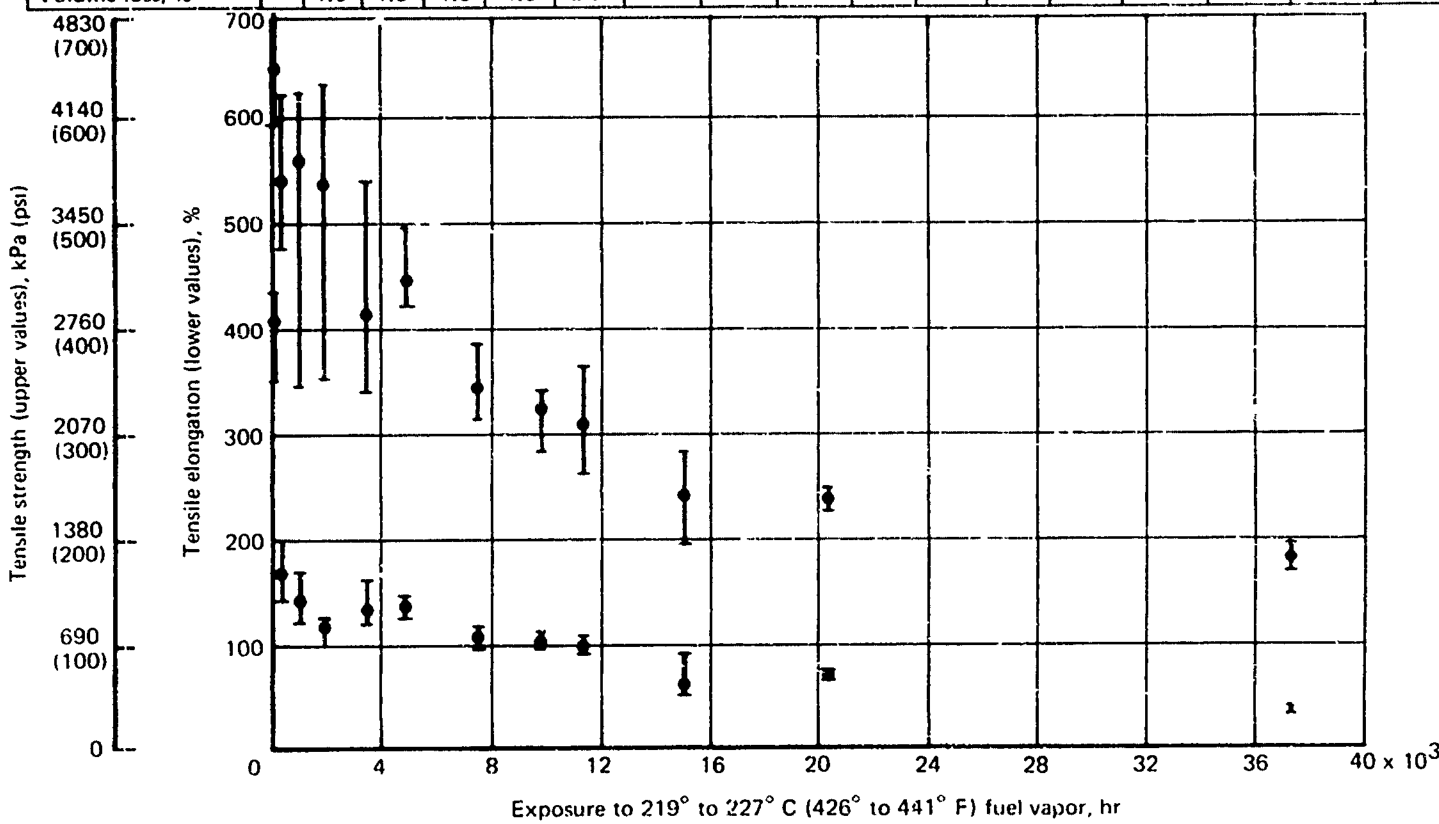


Figure 12.—Accelerated Cycle Exposure of DC77-028, Lot 1222, Tested at Room Temperature

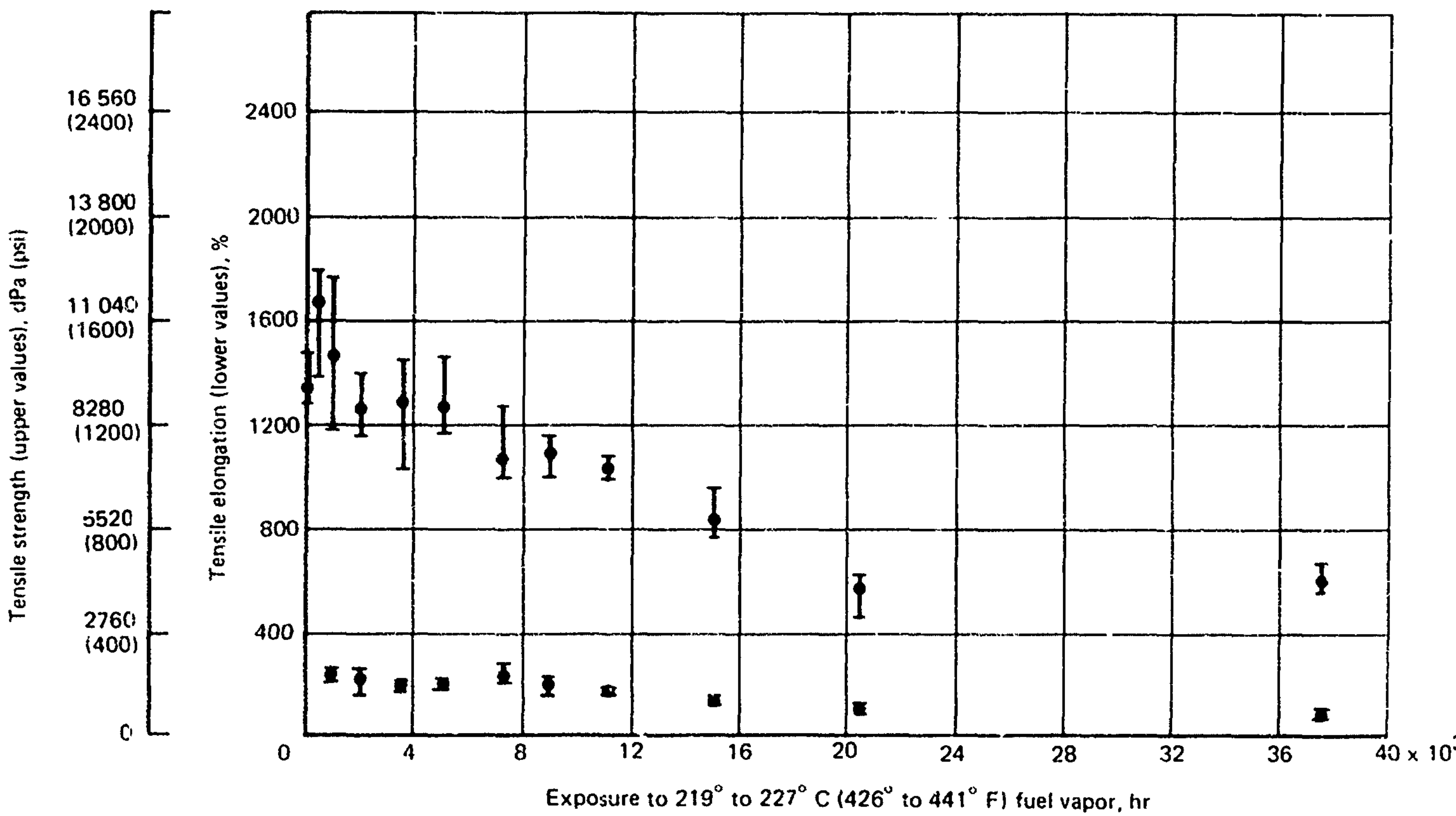


Figure 13.—Accelerated Cycle Exposure of DC 77-028 Lot 1222, Tested at -46° C (-50° F)

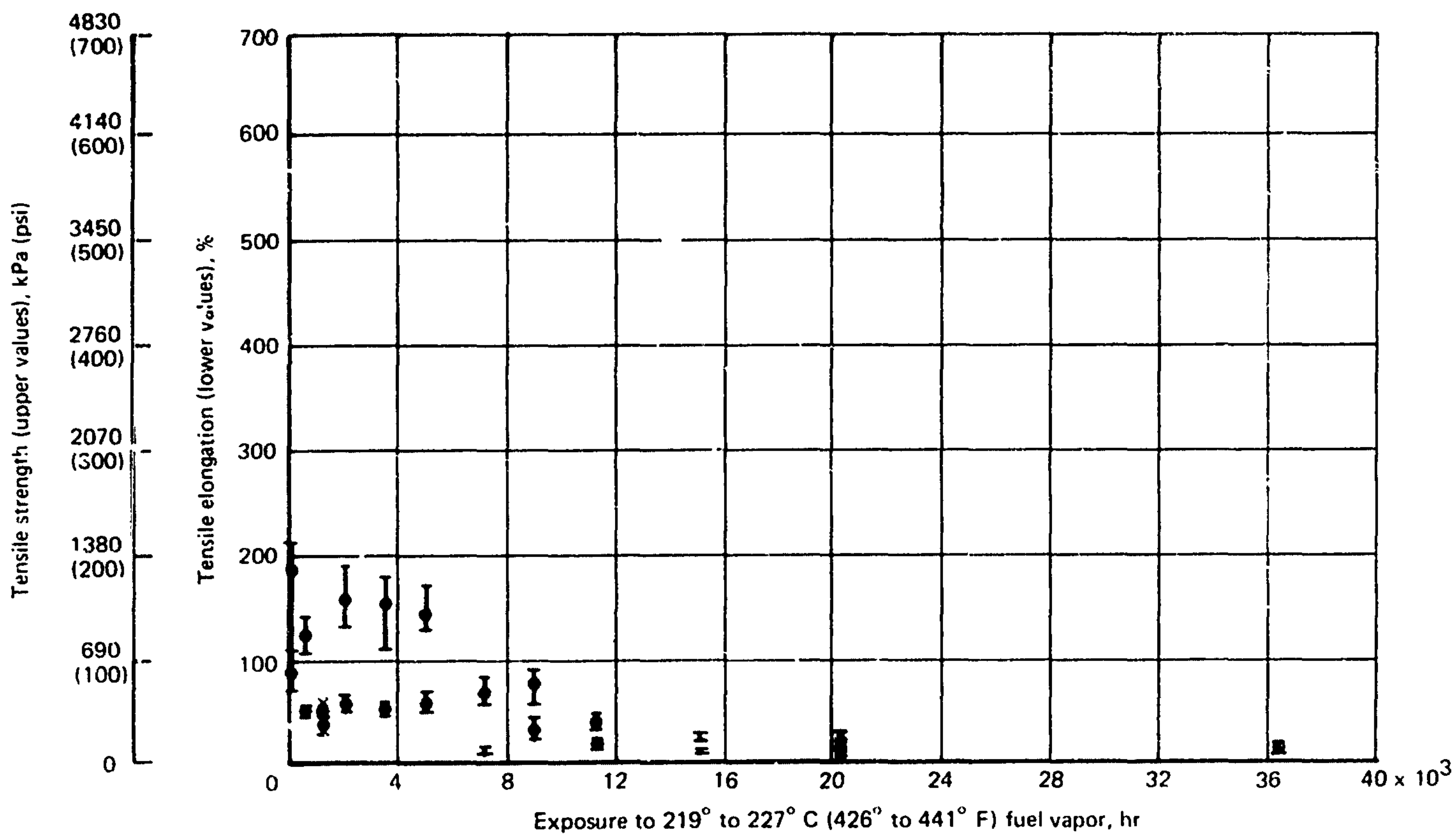
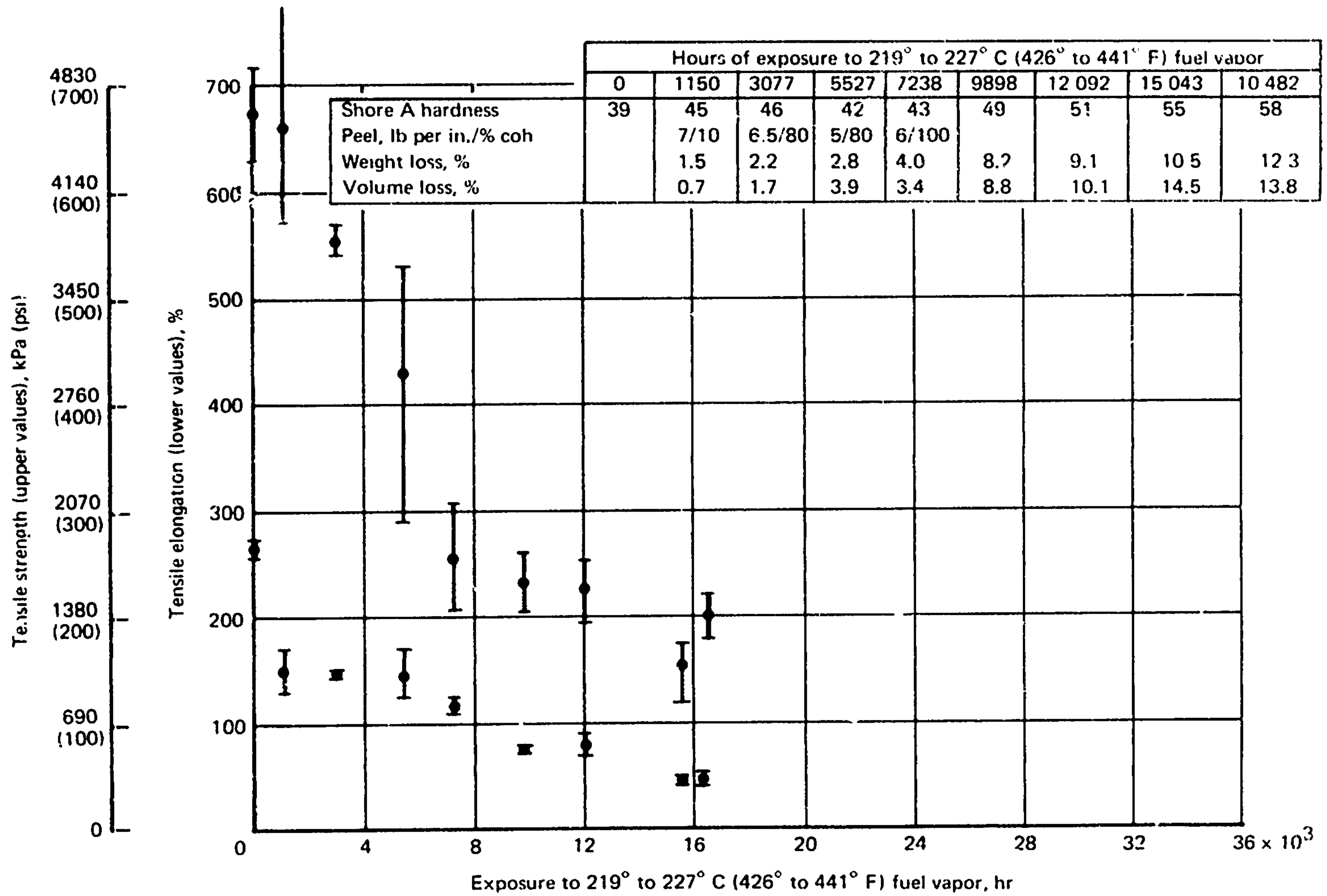


Figure 14.—Accelerated Cycle Exposure of DC 77-028, Lot 1222, Tested at 232° C (450° F)



	Hours of exposure to 219° to 227° C (426° to 441° F) fuel vapor								
	0	1150	3077	5527	7238	9898	12 092	15 043	10 482
Shore A hardness	39	45	46	42	43	49	51	55	58
Peel, lb per in./% coh		7/10	6.5/80	5/80	6/100				
Weight loss, %		1.5	2.2	2.8	4.0	8.2	9.1	10 5	12 3
Volume loss, %		0.7	1.7	3.9	3.4	8.8	10.1	14.5	13.8

Figure 15.—Specimens From Wichita Tank, Lot 205180, Tested at Room Temperature

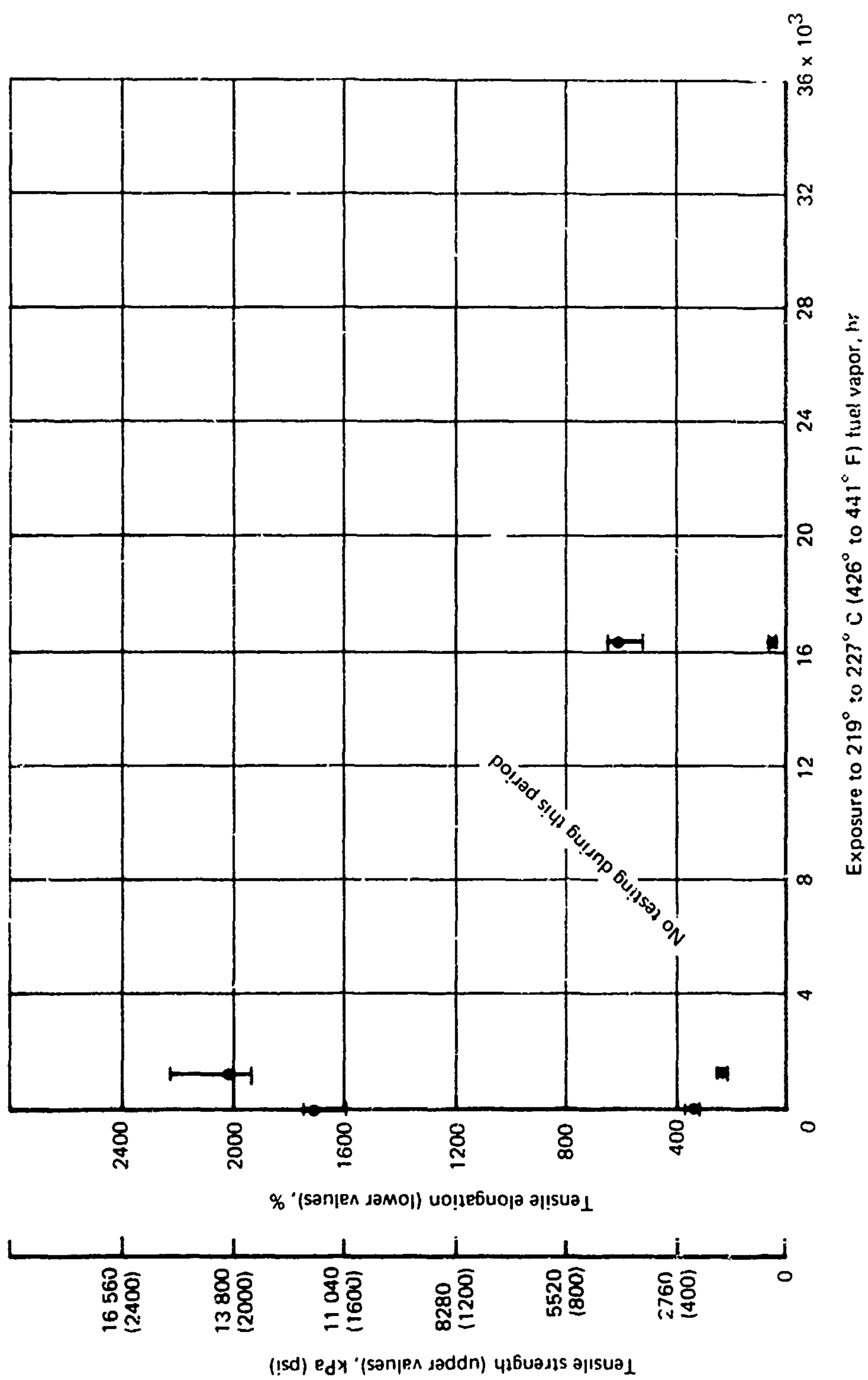


Figure 16.—Specimens From Wichita Tank, Lot 205180, Tested at -46° C (-50° F)

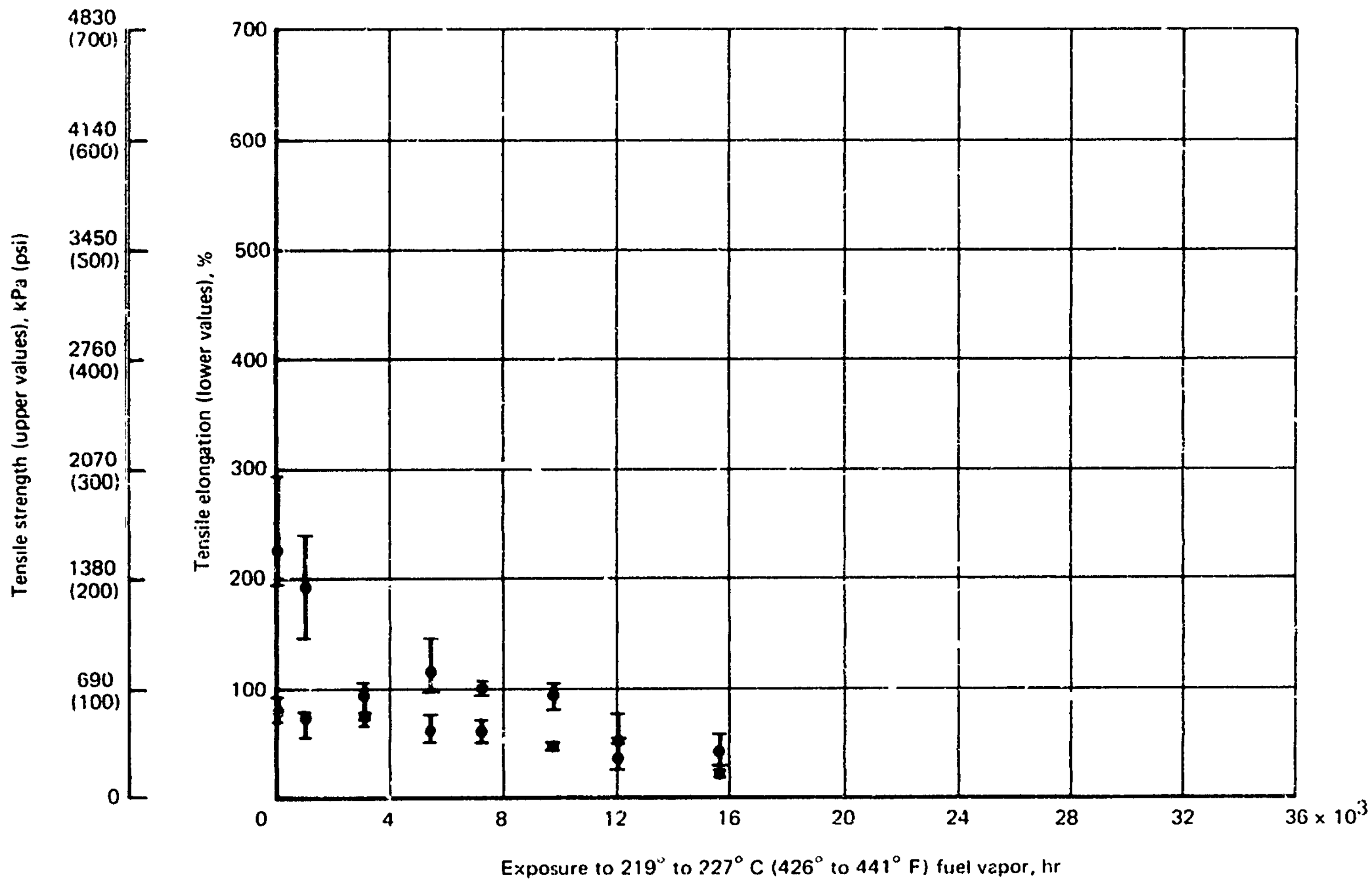


Figure 17.—Specimens From Wichita Tank, Lot 205180, Tested at 232° C (450° F)

	Hours of exposure to 219° to 227° C (426° to 441° F) fuel vapor											
	0	3012	5403	9335	5006	6872	18 618	20 174	22 460	22 598	24 662	27 828
Shore A hardness	33	36	41	42	43	42	45	44	44	44	41	51
Peel, lb per in./% coh												
Weight loss, %		3.0	4.8	6.3	8.6	8.4	9.6	10.2	10.4	9.6	10.0	13.3
Volume loss, %		3.2	5.2	6.7	8.0	8.6	10.ϵ	10.6	10.9	10.5	10.5	14 1

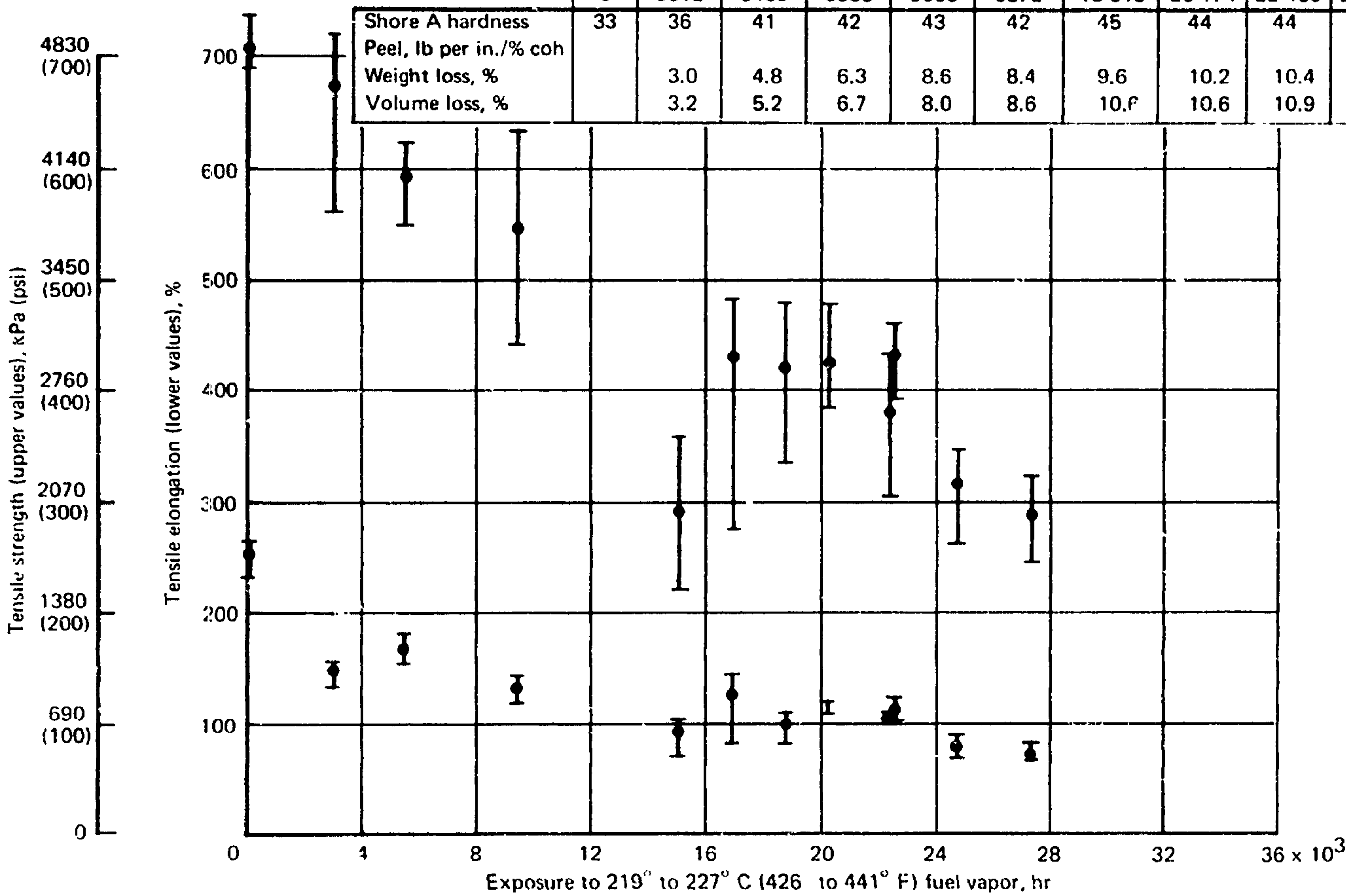


Figure 18.—Accelerated Cycle Exposure ^f DC 77-028, Lot 401117, Tested at Room Temperature

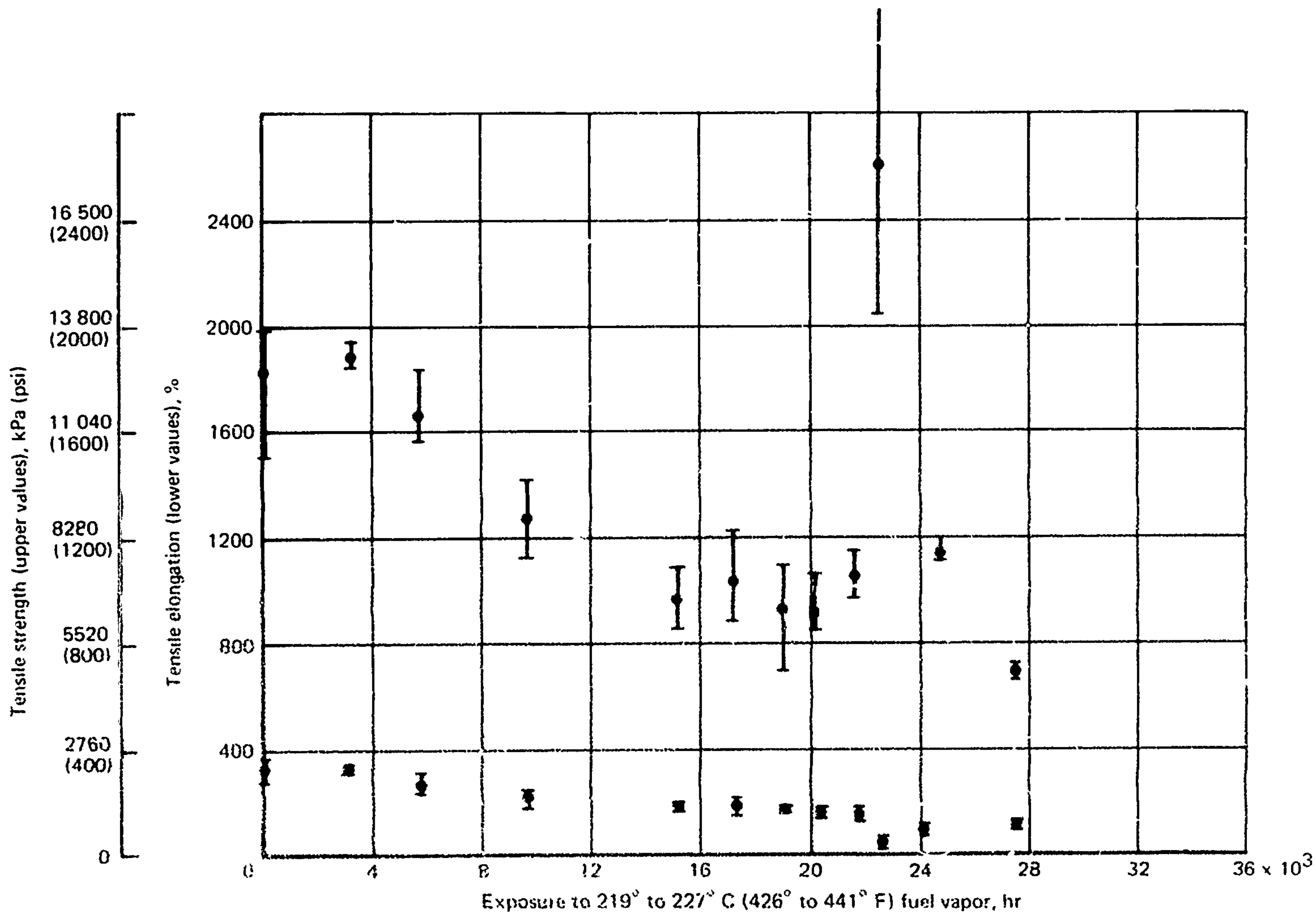


Figure 19.– Accelerated Cycle Exposure of DC 77-028, Lot 401117, Tested at -46° C (-50° F)

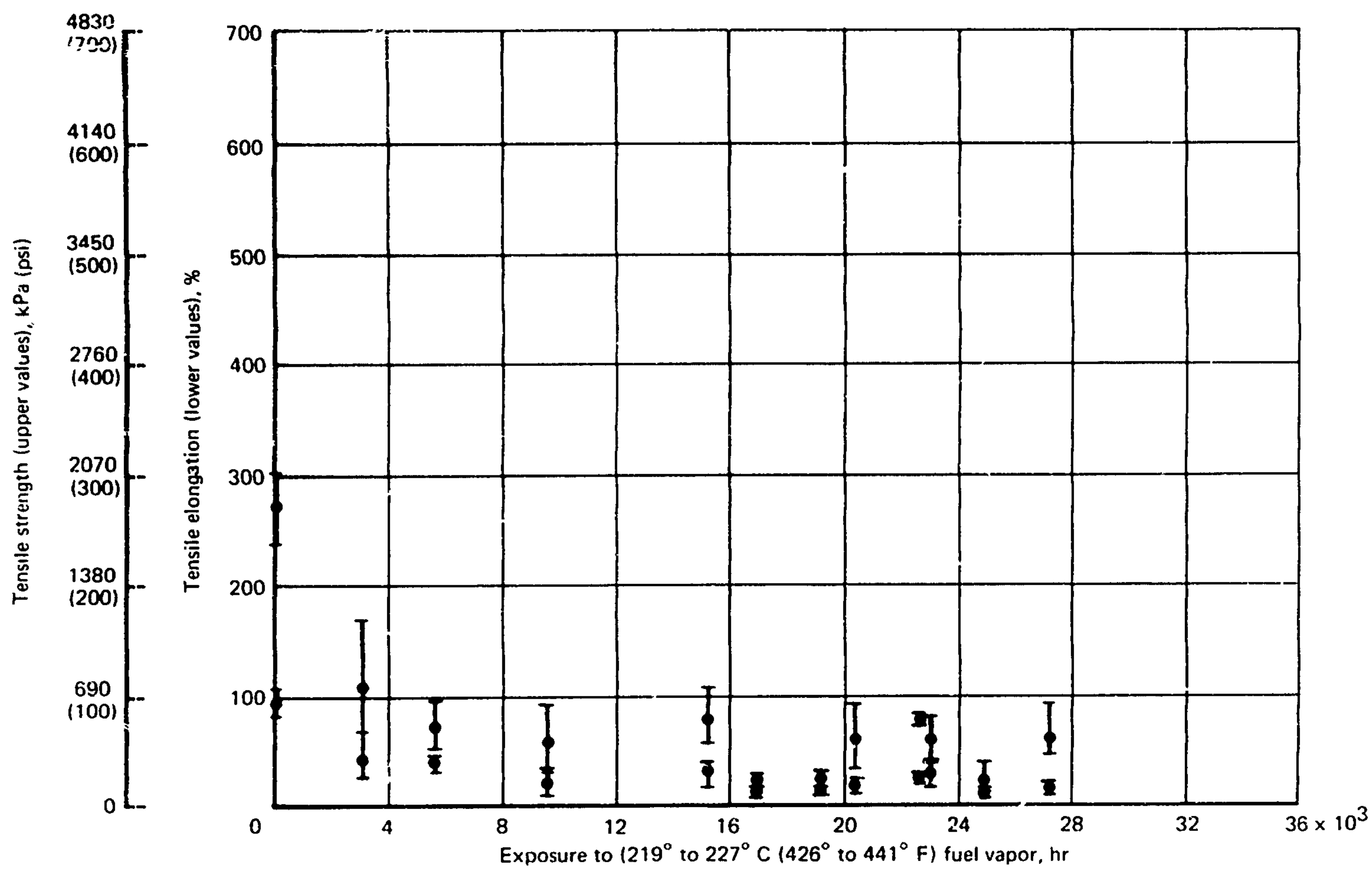
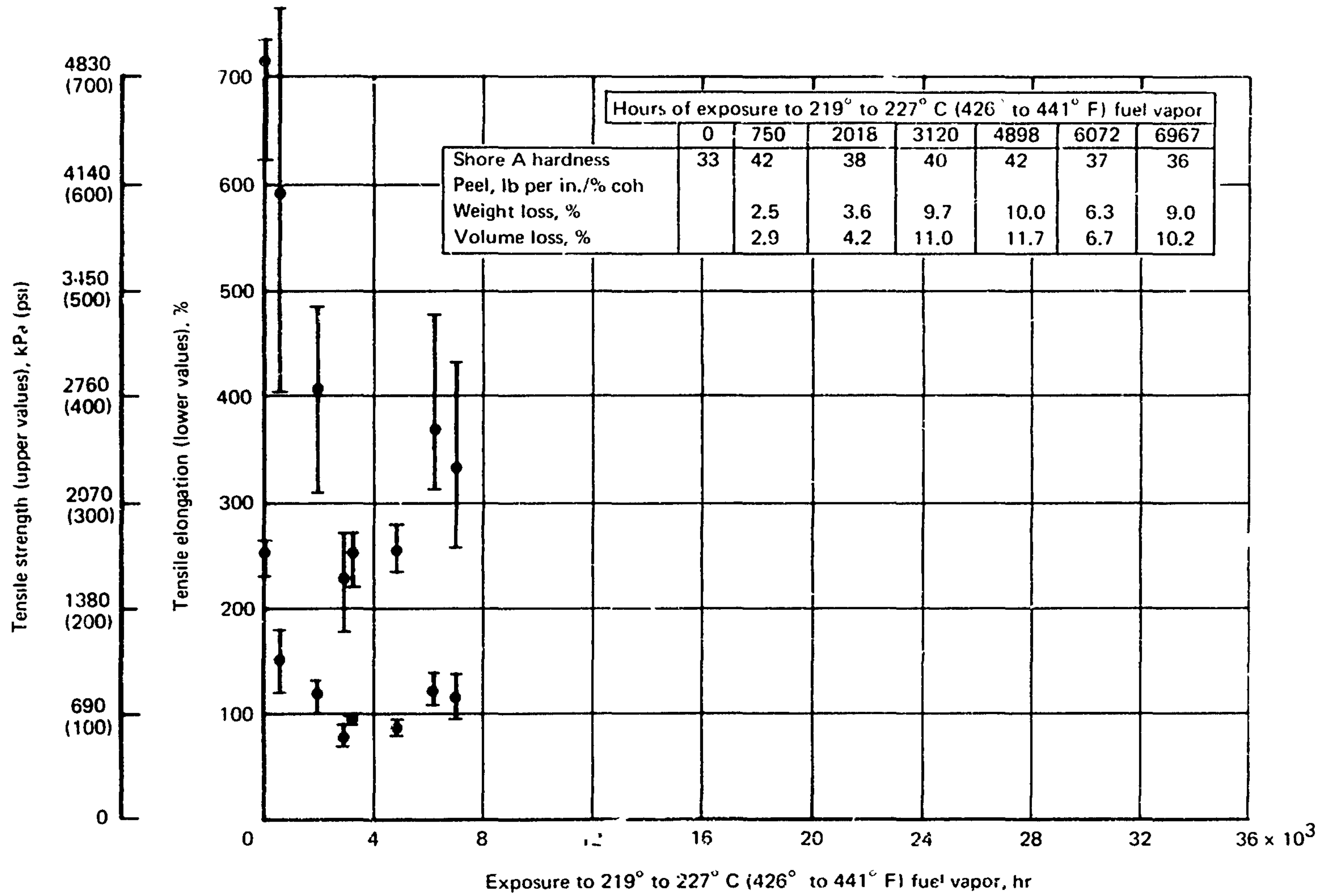


Figure 20.—Accelerated Cycle Exposure of DC77-028, Lot 401117, Tested at 232° C (450° F)



Hours of exposure to 219° to 227° C (426° to 441° F) fuel vapor	0	750	2018	3120	4898	6072	6967
Shore A hardness	33	42	38	40	42	37	36
Peel, lb per in./% coh							
Weight loss, %		2.5	3.6	9.7	10.0	6.3	9.0
Volume loss, %		2.9	4.2	11.0	11.7	6.7	10.2

Figure 21.—Flight Cycle Exposure of DC 77-028, Lot 401117, Tested at Room Temperature

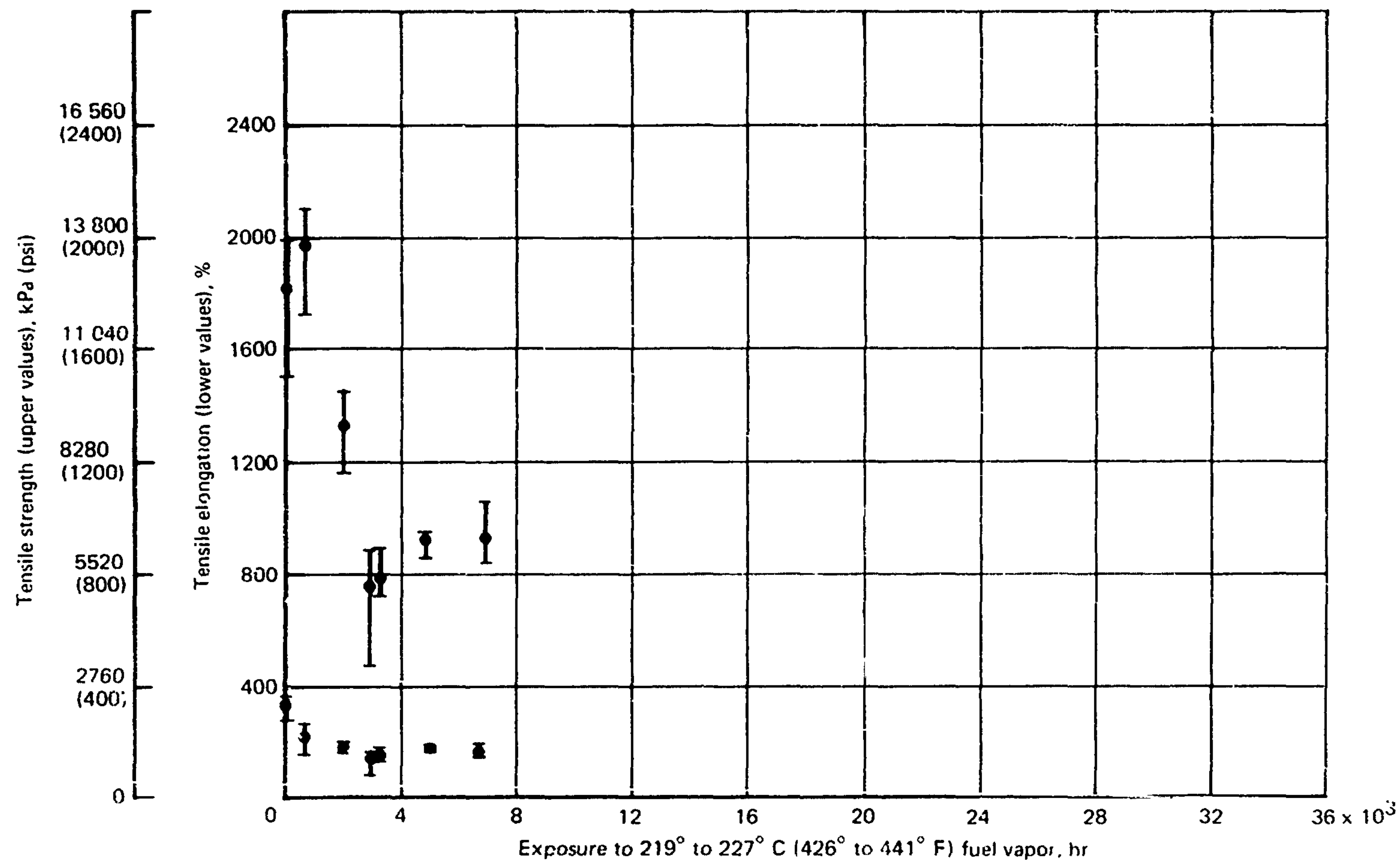


Figure 22.—Flight Cyc posure of DC 77-028, Lot 401117, Tested at -46° C (-50° F)

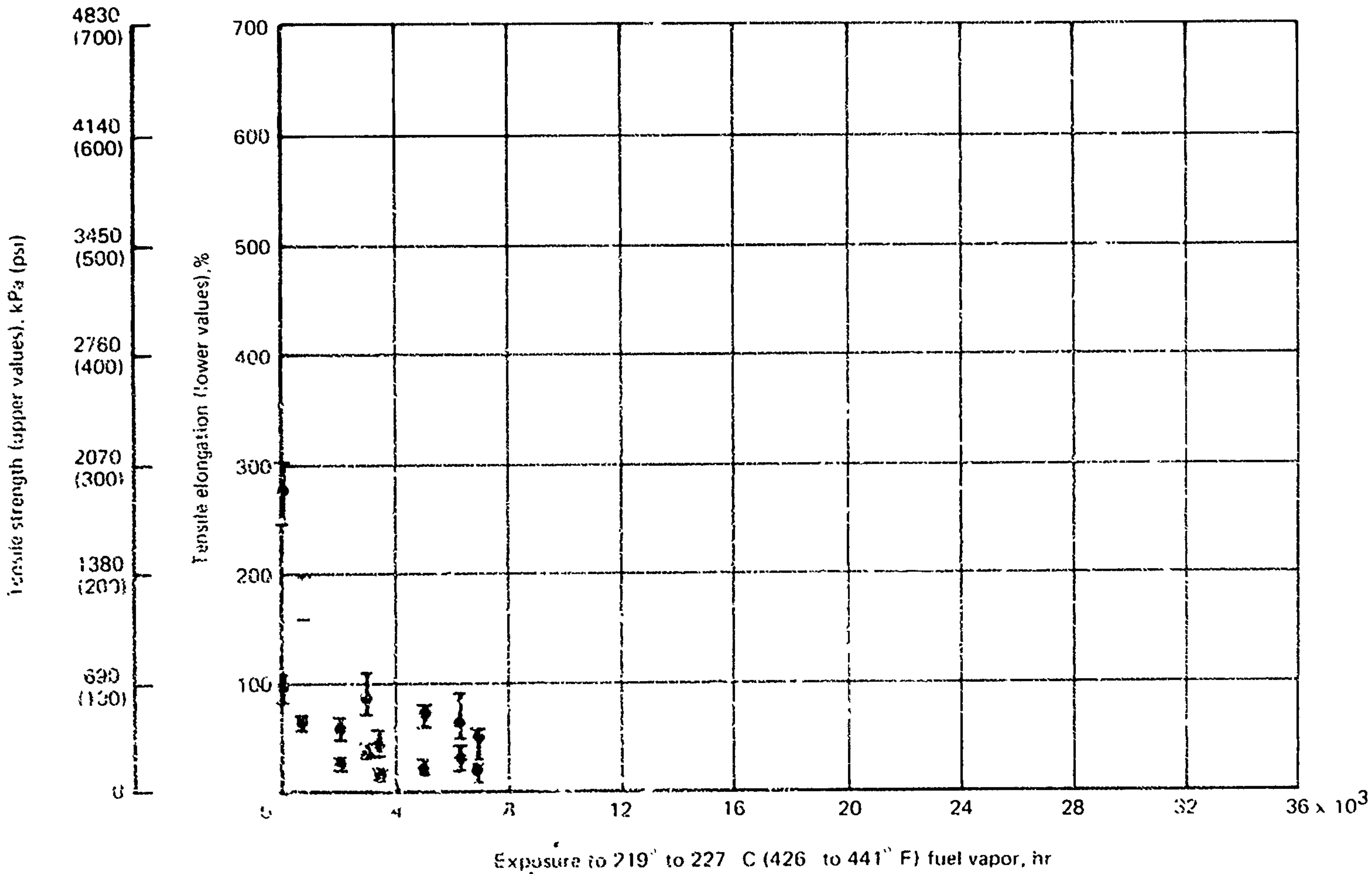
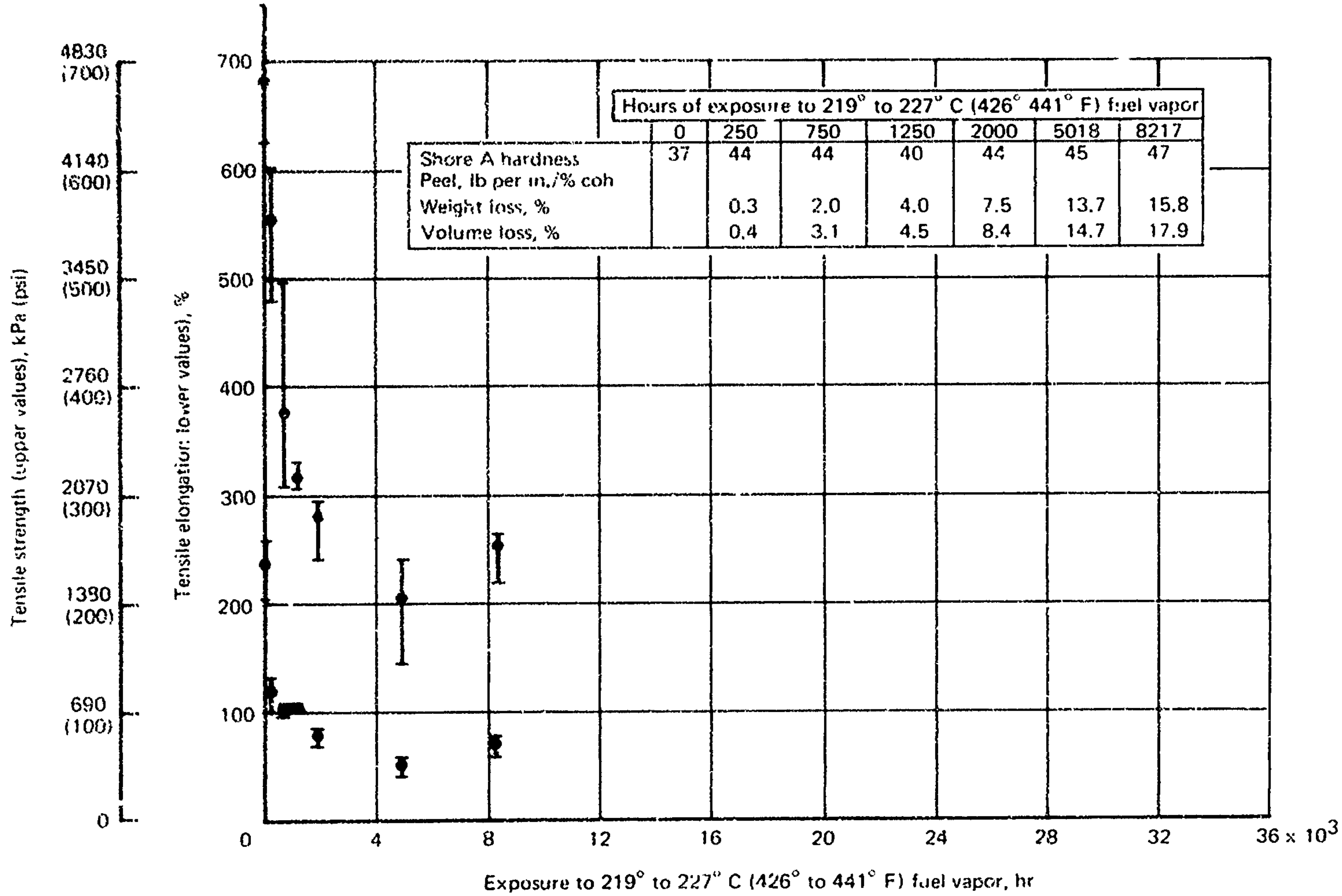


Figure 23.– Flight Cycle Exposure of DC 77-028, Lot 401117, Tested at 232° C (450° F)



	Hours of exposure to 219° to 227° C (426° 441° F) fuel vapor						
	0	250	750	1250	2000	5018	8217
Shore A hardness	37	44	44	40	44	45	47
Peel, lb per in./% coh							
Weight loss, %		0.3	2.0	4.0	7.5	13.7	15.8
Volume loss, %		0.4	3.1	4.5	8.4	14.7	17.9

Figure 24.—Flight Cycle Exposure of DC 77-028, Lot 206177, Tested at Room Temperature

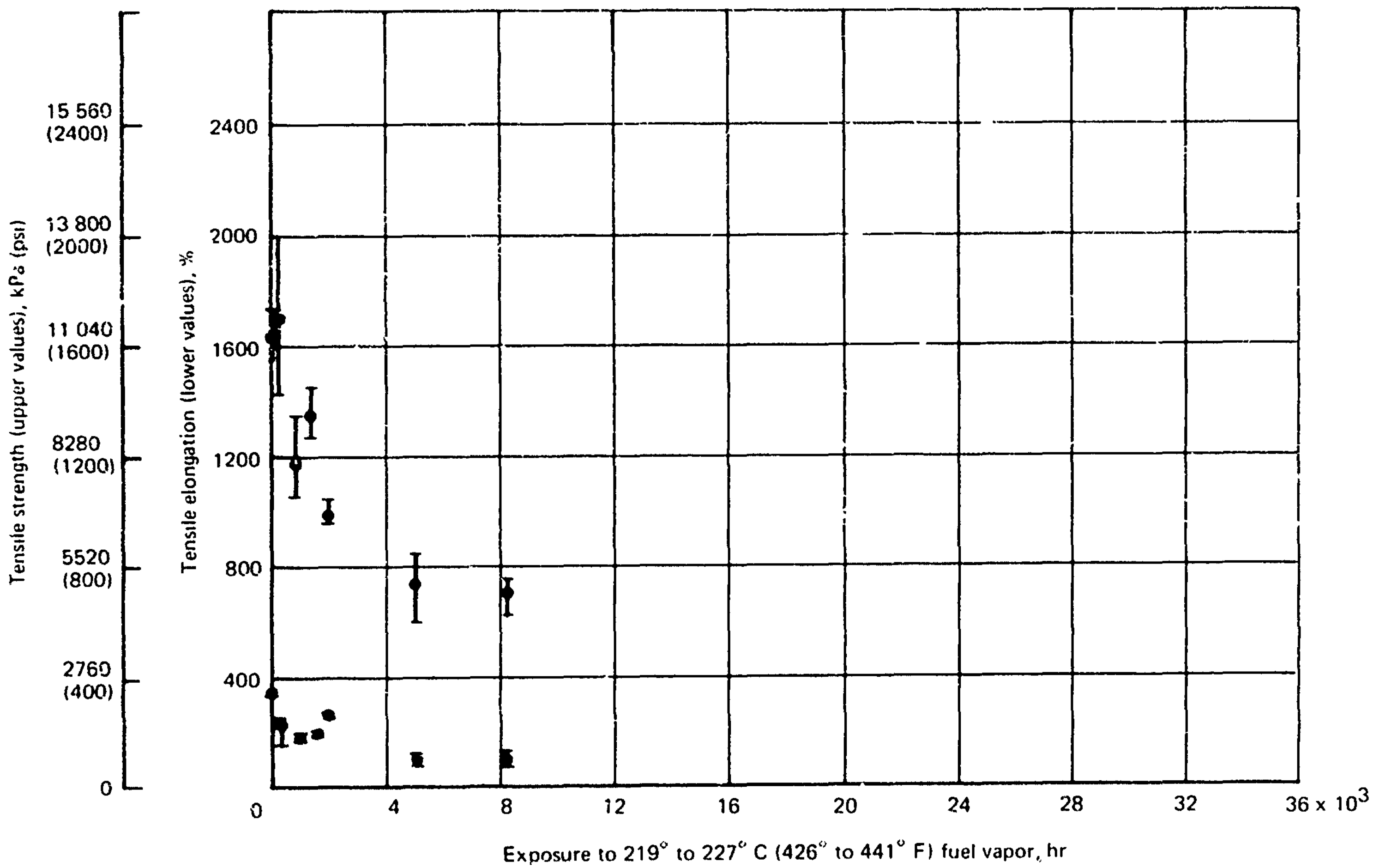


Figure 25.—Flight Cycle Exposure of DC 77-028, Lot 206177, Tested at -46° C (-50° F)

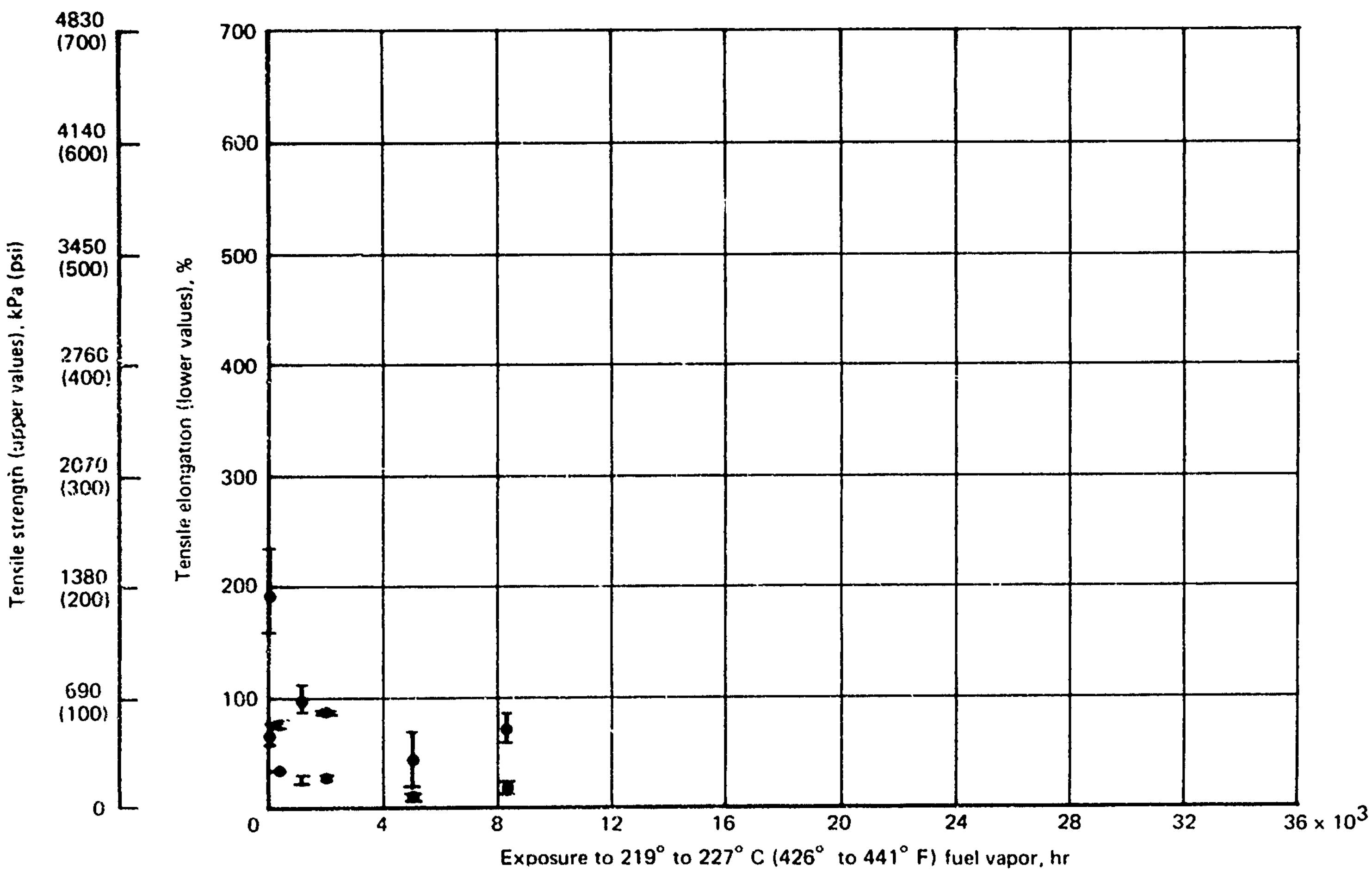


Figure 26. –Flight Cycle Exposure of DC 77-028, Lot 206177, Tested at 232° C (450° F)

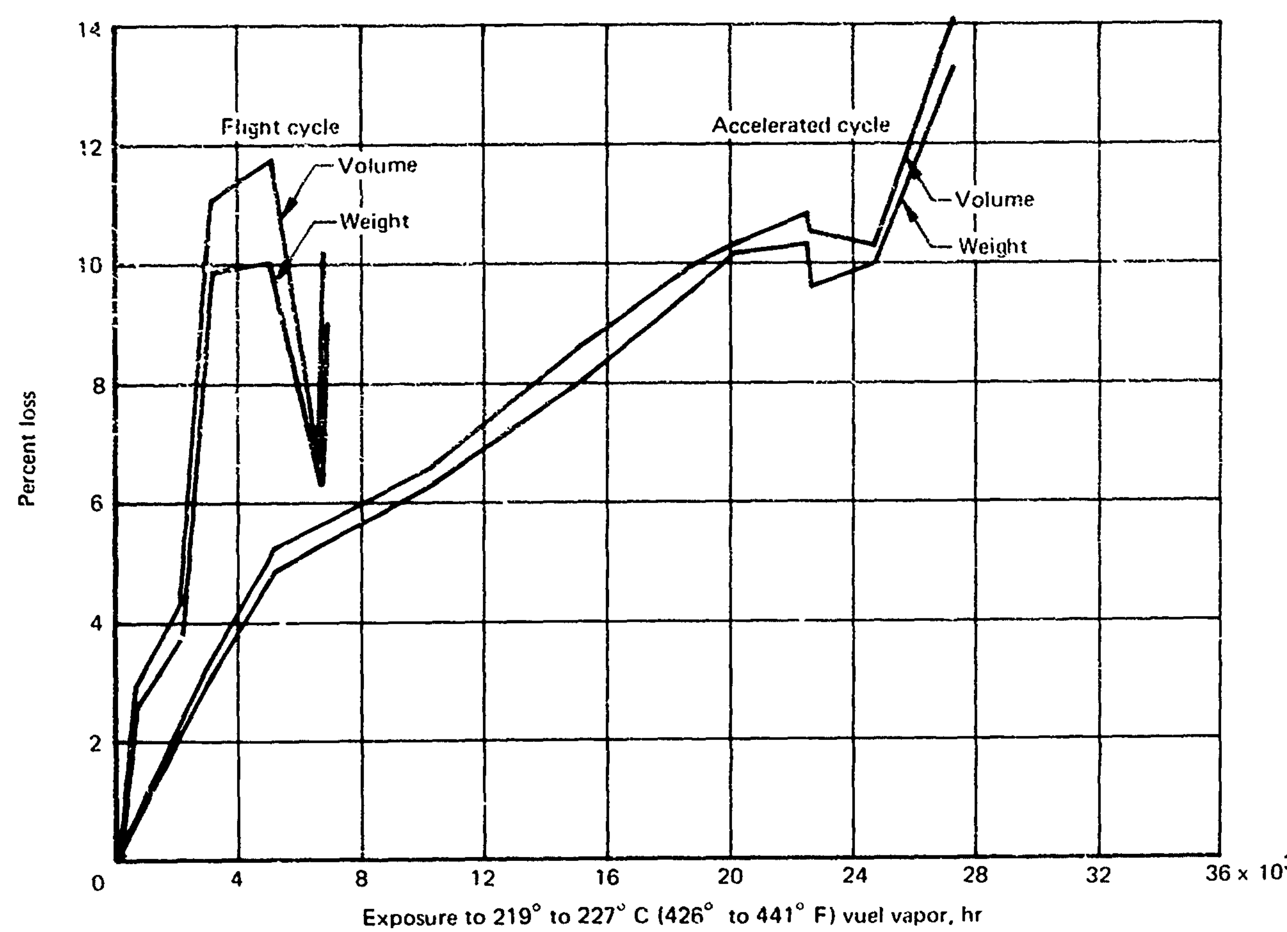


Figure 27.—Weight and Volume Loss for DC 77-028, Lot 401117

Figure 28.—Fillet Seal of Picture Frame Panel After Exposure

remained essentially leak-free. The faying surface sealed panel was leaking slightly. Based on this and the fact that other tests showed that the faying surface sealant is not effective, we must assume that the test is unreliable.

3.2.2 FILLET DEFLECTION

After 6967 hr of 219° to 227° C (426° to 441° F) fuel vapor exposure and 836 040 load cycles in the flight cycle, only small cracks were apparent at the juncture of each fillet and the titanium overlap. By visual inspection, these cracks did not show evidence of growing after they were noted initially.

3.2.3 TANK TESTING

The tank test is considered to be the most informative in regard to sealant performance in an airplane structure. The tank is shown in figure 29 after 6000 load cycles at room temperature, 4500 cycles at 232° C (450° F), 4330 cycles at -46° C (-50° F), and exposure to fuel and fuel vapor for the temperature and hours indicated in table 1. The tank was inspected and tested for leaks once every 6 months. The leaks varied each time in number and location. Detection of leaks was never possible without pressurization. For the final test, the tank was left to stand for about 1 hr with water in it and it did not leak. Approximately 13.8-kPa (2-psi) pressure was then applied, which caused two spots to leak very slightly. After a short period of time, oozing was noted at one other location. Pressure of 34.5 kPa (5 psi) was then applied and the leaks increased in amount and number. When the pressure was removed, the tank continued to leak. It was then drained.

Table 1.—Exposure Times for the Test Tank

Temperature, °C (°F)	Hours
Liquid	
To 59 (139)	676
60 (140) to 65 (150)	2 133.5
Over 65 (150)	113.2
Below 60 (140)	521.5
Vapor	
60 (140) to 204 (400)	1 794.8
205 (401) to 218 (425)	470
213 (415) to 227 (441)	78.5
219 (426) to 227 (441)	16 481.5
Over 227 (441)*	65

*Included in hours at 219° to 227° C (426° to 441° F); highest was 238° C (460° F).

It had been noted previously that cracks in the sealant, such as those shown in figure 30, were more prevalent when the sealant fillet was thick, so a small section was

Figure 29.– Test Tank

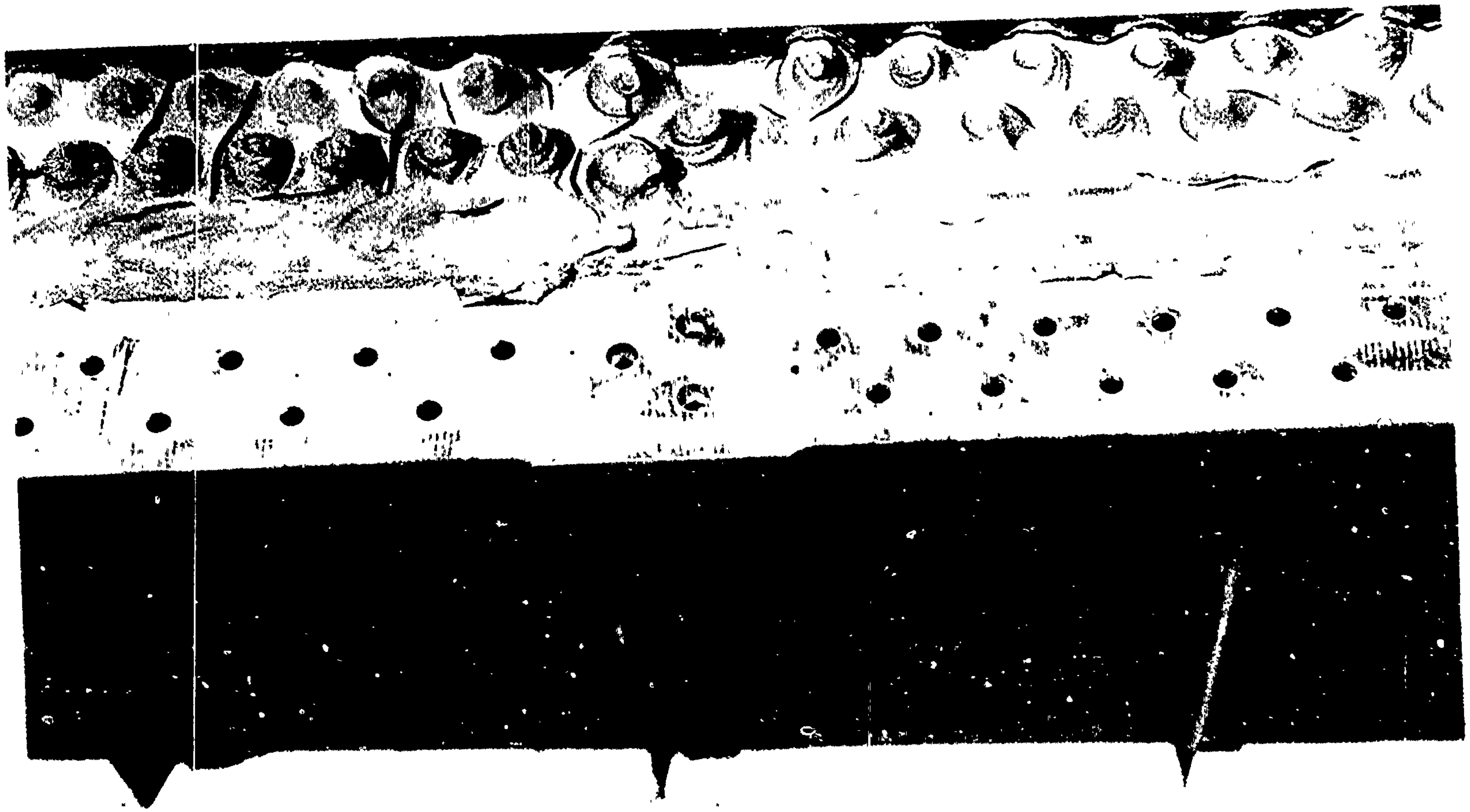


Figure 30.--Sealant Condition After Exposure

repaired using small fillets. The section was repaired after 7238 hr of 219° to 227° C (426° to 441° F) fuel vapor exposure in February 1973 and had no splits and no leaks. Adhesion was retained to the old sealant as shown in figure 31.

The top and bottom skins were removed. Adhesion of the sealant to the fasteners and structure was surprisingly good, and the sealant fillets failed cohesively when the structural components were separated, as can be seen in the pictures. Areas where the faying surface isolation seals had been were evident (figs. 32 and 33), but there was little sealant to be seen. What was there was easily scored with the blunt edge of a fingernail. Condition of the Teflon rings in the self-sealing fasteners (which were unsuccessful from the beginning) varied from no change through a crumbly powder to nonexistent. The sealant on the top skin had separated from the seal plane, had numerous large splits, and was absent in some places.

Each inspection revealed leaking corners which were usually repaired before returning the tank to environmental exposure. The injection sealant was inadequate in that it expanded, gradually extruded out of the groove, and ruptured the adjacent fillet. Modification of the DC 77-028 fillet sealant to produce DC 77-066 injection sealant was demonstrated to be partially effective. At one time, corner repairs were made using DC 77-028; the corners started leaking badly immediately when exposed to temperature. The injection seal is shown in figure 34.

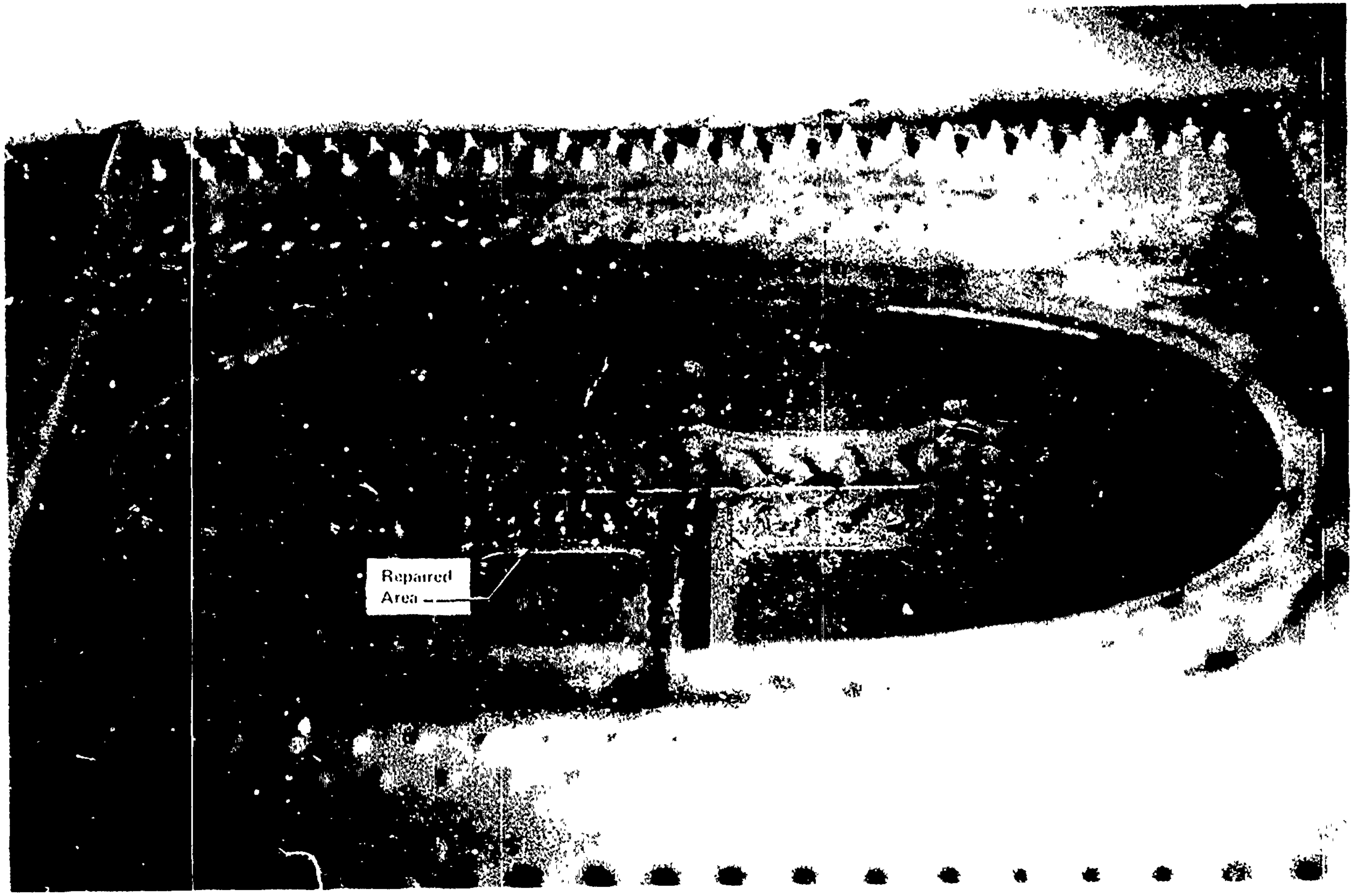


Figure 31.–Repaired Area

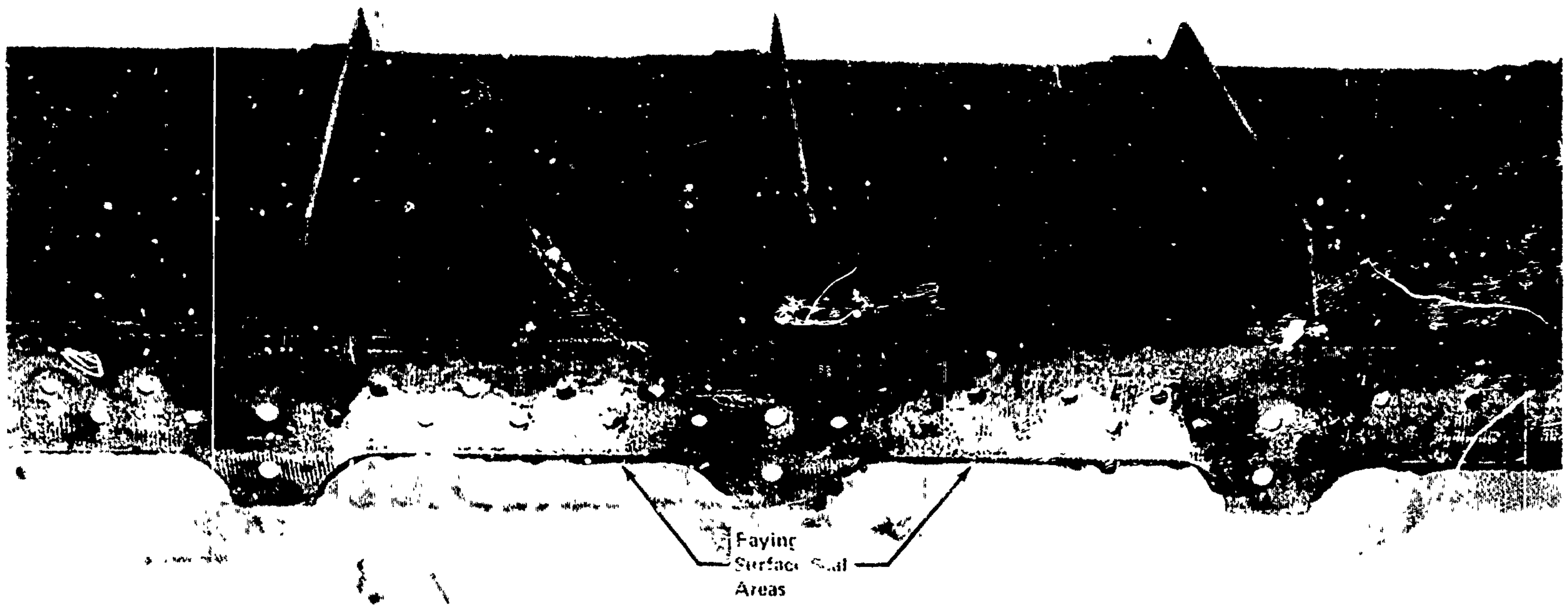


Figure 32.—Lower Web-to-Chord Faying Seal Location

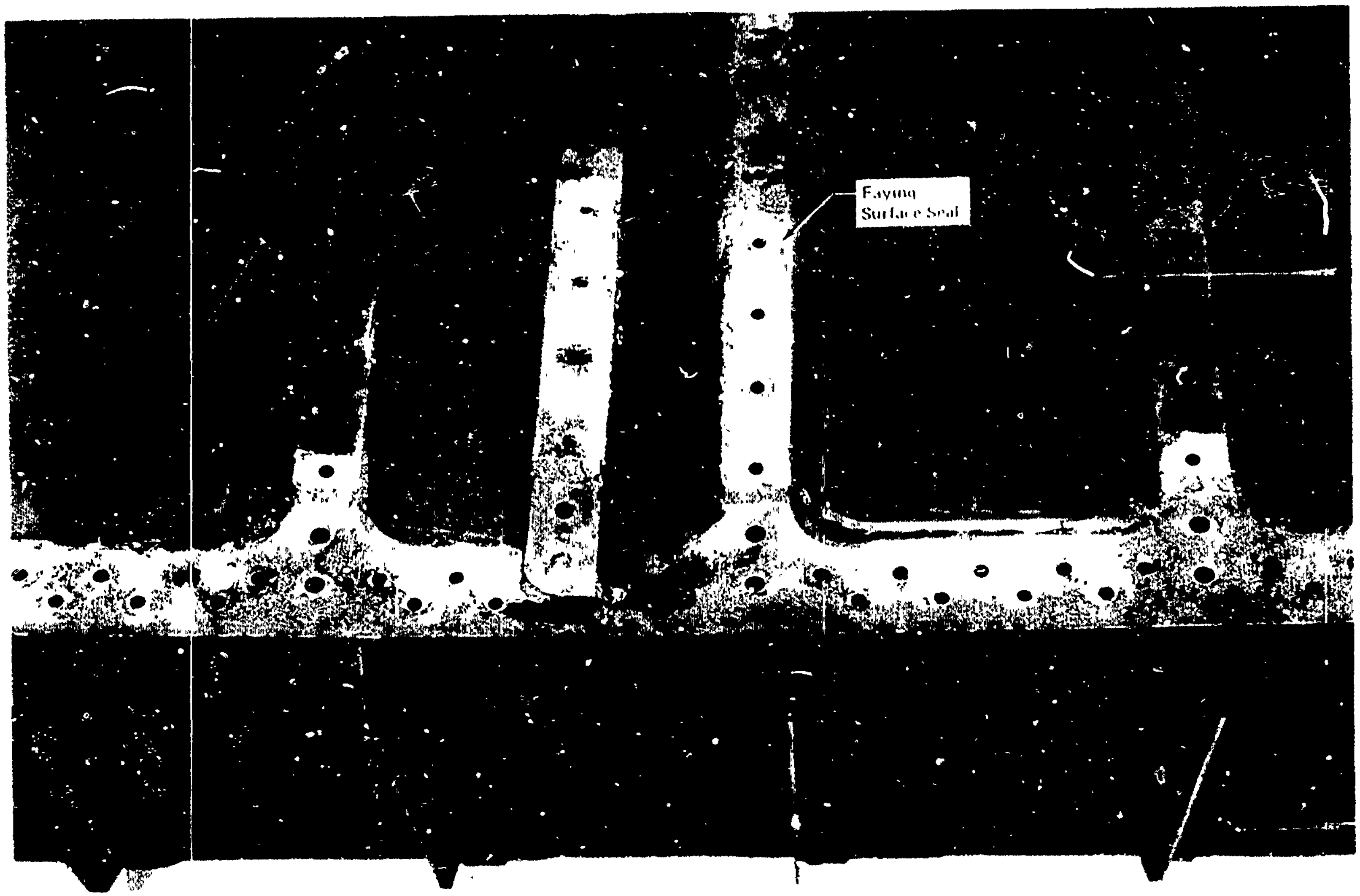


Figure 33.--Stiffener-to-Web Faying Seal Location

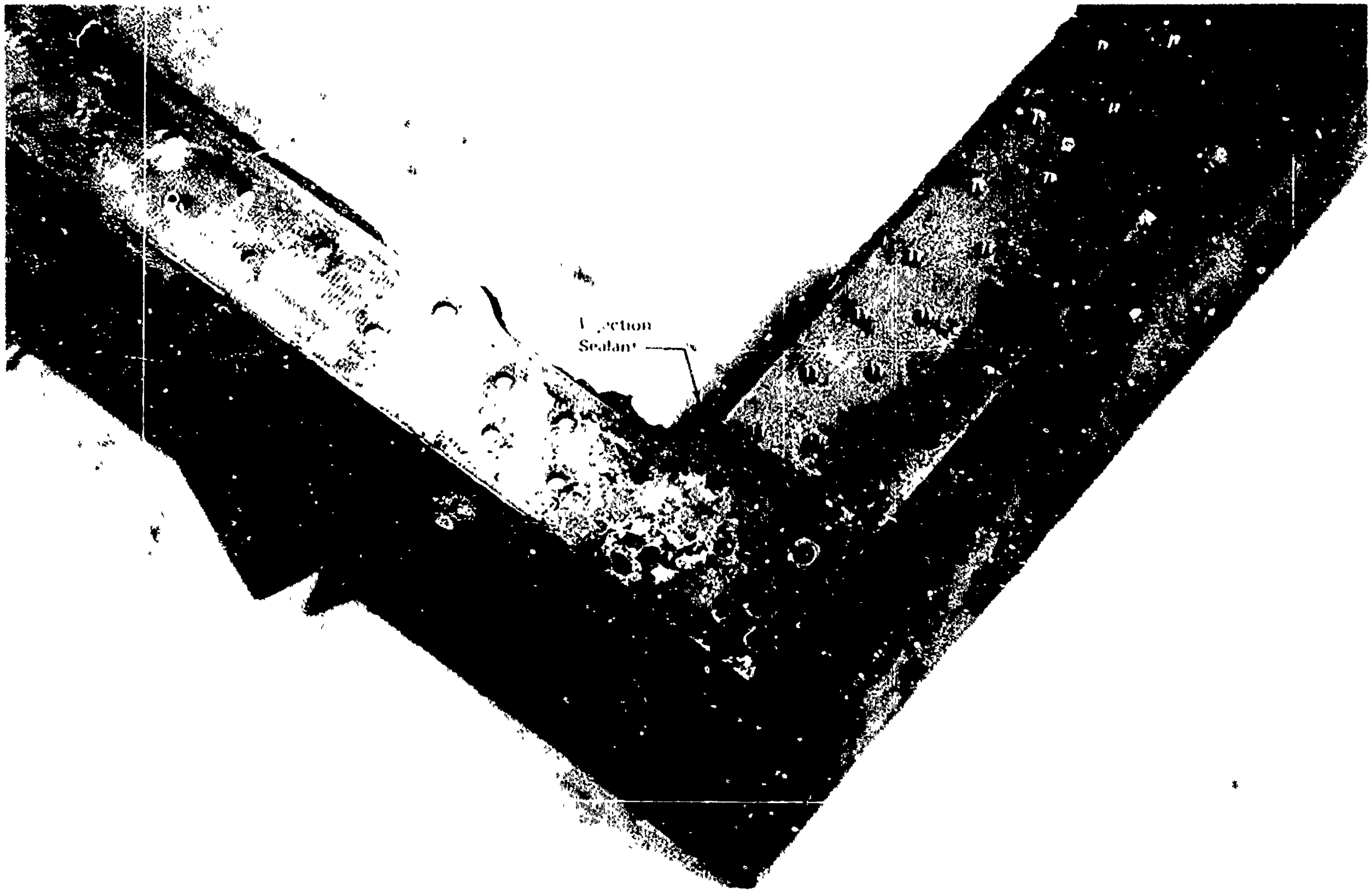


Figure 34.—Injection Seal

4.0 BACKUP SEALANT

4.1 HYDROFLUOROCARBON

The Products Research and Chemical Corporation (PRC) was given a contract to develop a sealant based on fluorocarbons and having a high solids content. Existing sealants based on fluorocarbons are normally supplied with a maximum of 40% solids by volume, requiring them to be applied in thin cross sections and resulting in shrinkage on drying and curing. PRC was asked to develop one having a solids content of 80% by volume; one having 70% was produced, which is considered a significant advancement. Solvent content was further reduced to less than 10% by volume by pre-extruding into a tape which was tacky enough to adhere to titanium. After a heat cure, adhesion was retained; however, when an attempt was made to cure a sheet of sufficient thickness to provide test specimens, bubbling took place and destroyed the sheet. The progress made was encouraging and further investigation and development are warranted, specifically in adhesion, thermal stability, and stress corrosion. A complete report of the development is attached as appendix B.

4.2 HYBRID FLUOROCARBON-FLUOROSILICONE

The Air Force Materials Laboratory funded the development of hybrid fluorocarbon-fluorosilicones by the Dow Corning Corporation. One of the most promising of these, DC 77-108 (FCS 210), was exposed to accelerated cycling and tested periodically. Test results are shown in table 2.

Table 2. -DC 77-108 Test Results

Property	Hours of 219° to 227° C (426° to 441° F) fuel vapor exposure			
	0	2520	9938	15 826
Tensile strength, kPa (psi)				
-46° C (-50° F)	24 019 (3486)	17 749 (2576)	19 120 (2775)	8342 (1209)
Room temperature	7 400 (1074)	7 917 (1149)	7 237 (1059)	4885 (708)
232° C (450° F)	875 (127)	1 523 (221)	1 054 (153)	849 (123)
Elongation, %				
-46° C (-50° F)	8	17	7	8
Room temperature	585	640	270	190
232° C (450° F)	64	111	44	26
Shore A durometer (hardness)	34	45	76	46
Weight loss, %		7.0	7.4	7.4
Volume loss, %		6.8	7.5	7.3

5.0 CONCLUSIONS

1. Confidence was developed that the Dow Corning DC 77-028 (DC 94-529) fluorosilicone sealant system would perform for 50 000 flight-hours as an integral fuel tank fillet sealant in a commercial supersonic airplane. This confidence was established by the completion of the following evaluations:

   a. More than 36 000-hr accelerated life cycle test in 219° to 227° C (426° to 441° F) fuel vapor

   b. More than 8000 flight cycles test (24 000 hr) in 219° to 227° C (426° to 441° F) fuel vapor under ±96.6-kPa (±14-psi) pressure variance

   c. More than 16 000 hr of test as sealant in a simulated SST fuel tank under accelerated life cycle conditions in 219° to 227° C (426° to 441° F) fuel vapor

   d. Torsional load cycle tests of the sealed tank (item c) of 4330 at -46° C (-50° F), 6000 at room temperature, and 4500 at 232° C (450° F)

   e. Demonstration of fillet sealant repairability during tank tests

   f. Demonstration of lot-to-lot reproducibility of production size lots of DC 77-028

2. Neither the faying surface sealant (DC 77-053) nor the injection sealant (DC 77-066) derivatives of the DC 77-028 fluorosilicone system were satisfactory; however, the DC 77-066 was superior to DC 77-028 as an injection sealant.

3. The backup fluorocarbon and hybrid fluorocarbon-fluorosilicone sealant systems show promise but do not have the low-temperature ductility capabilities demonstrated by the fluorosilicone sealant.

4. Flight cycle testing provides more critical evaluation of basic sealant properties for a given total test time than accelerated life cycle testing. Testing of the sealant in its intended application in a simulated fuel tank is most informative from a practical standpoint of sealant application, behavior, and repairability.

6.0 RECOMMENDATIONS

Further development of a faying surface and injection sealant should be pursued.

Flight testing of the DC 77-028 system in a supersonic aircraft is the next logical step in investigation of its functionality and should be initiated.

Attempts should be made to develop fuel tank sealants from polymers other than fluorosilicones, especially if aircraft designed to fly in excess of Mach 2.7 are contemplated. These sealants should be tested in the same way as DC 77-028 for comparison purposes.

APPENDIX A

THE BOEING COMPANY
WICHITA DIVISION

CODE IDENT. NO. 81205

NUMBER D3-8297 REV LTR F

INITIAL RELEASE DATE APR 13 1970

TITLE EVALUATION OF THE PROPOSED SST FUEL TANK SEALANT IN SMALL SIMULATED FUEL TANKS

FOR LIMITATIONS IMPOSED ON THE USE OF THE INFORMATION CONTAINED IN THIS DOCUMENT AND ON THE DISTRIBUTION OF THIS DOCUMENT, SEE LIMITATIONS SHEET.

MODEL 2707 CONTRACT

ISSUE NO. ISSUED TO

PREPARED BY Lyle Middleton 3-31-70

SUPERVISED BY Charles E Johnson 4-1-70

APPROVED BY R A Ballweg 4/2/70

APPROVED BY [illegible] 4/3/70

E-3039 R4

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ABSTRACT

In order to determine the fuel tank sealing capabilities of the sealant (Dow Corning 77-028) proposed for the fuel tanks of the Model 2707 supersonic transport prototypes, two small titanium test tanks were initially fabricated and sealed at Boeing-Wichita. These two tanks were subjected to long term simulated fuel tank environments and were periodically subjected to cyclic structural loading at three temperatures, followed by pressure testing for leakage. A third test tank, which more closely simulated the SST fuel tank structure, was subsequently fabricated and sealed by CAG. This tank was exposed to similar environmental conditioning and structural loading.

Testing of tank numbers 1 and 2 is described in Section 2. Testing of tank no. 2 was terminated after 524 hours of fuel vapor exposure at 415 - 441°F (4 weekly environmental conditioning cycles). Testing of tank no. 1 continued to 3862 hours of fuel vapor exposure (31 environmental conditioning cycles).

Testing of tank number 3 is described in Section 3. This tank was exposed to a total of 16,482 hours of fuel vapor at 426 - 441°F or a total of 22,269 hours at all conditions (liquid fuel, ambient to 205°F, and vapor fuel, ambient to 460°F). Total number of weekly environmental conditioning cycles was 129. In addition the tank was periodically load cycled for an accumulated 6000 cycles at ambient temperatures, 4500 cycles at 426 - 441°F, and 4330 cycles at -40 to -50°F.

Early in the environmental conditioning of all three tanks the sealant exhibited numerous cracks and areas of loss of adhesion resulting in significant leakage. This document describes in detail the environmental conditioning and structural loading used, and the condition of the sealant and the observed leakage of the tanks after each increment of specified testing. However, all conclusions concerning the significance of the testing and the anticipated performance of the sealant will be presented elsewhere by the Commercial Airplane Group, and is therefore beyond the scope of Wichita Division responsibility.

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FOREWORD

This report was prepared by the Materials Technology Unit of The Boeing Company, Wichita Division. Work was initiated under Engineering Work Authorization Number 36529221, titled "Environmental and Functional Testing Of SST Fuel Tank Sealant", dated August 4, 1969 (written by the Commercial Airplane Group, Structures Technology-Materials-SST).

On March 25, 1971 this program was cancelled when the government denied additional funds for SST prototype construction.

Work was subsequently resumed in August 1971 under Commercial Group IDWA No. 630600 (EWA No. 964020), and Wichita EWI No. 964020 funded by the Department of Transportation.

Funding was continued in April 1972 under Commercial Airplane Group IDWA No. 630600 (EWA No. 03866), and Wichita EWI No. 989001.

In December 1973 the final portion of the program was authorized under Commercial Airplane Group IDWA No. B30600 and Wichita EWI No. 989001-1.

The program was completed in March 1975.

1. INTRODUCTION

The most promising sealant available to date for sealing the fuel tanks of the Model 2707 Supersonic Transport is Dow Corning 77-028, a two-part experimental fluorosilicone, which was developed specifically for this purpose. This sealant must function for at least 50,000 hours in the presence of liquid hydrocarbon fuel up to 150°F and fuel vapor and nitrogen up to 441°F.

The purpose of the work conducted herein is to verify the sealing capabilities of this sealant by environmental and structural load testing of small sealed titanium test tanks.

To accomplish the above objective two small titanium tanks were initially fabricated and sealed. These tanks simulate typical skin and stiffener type integral fuel tank structure. One tank was fillet sealed only and the other was fillet sealed and faying surface sealed. These tanks were exposed to long term fuel tank environments, and were periodically subjected to structural dynamic torsional loading, followed by pressure testing, to determine the capability of the sealant to retain fuel at the environmental temperature extremes of the SST fuel system.

A third tank was subsequently built by CAG and tested by Wichita. This tank more closely simulated the SST fuel tank structure than did the first two tanks.

Laboratory test specimens were prepared and installed in each tank. These specimens were environmentally conditioned in the tanks and were withdrawn periodically and evaluated to measure the change in physical properties.

2. TESTING OF TANK NUMBERS 1 AND 2

2.1 Fabrication and Sealing

1. The test tank design used herein was originally developed by Boeing-Wichita under a contract with the Air Force Materials Laboratory (AFML). Reference 1 is the final report under this contract. The detail parts for two test tanks, which were originally fabricated for this contract but not used, were returned to Boeing-Wichita by AFML and used to build the two test tanks described herein. Optimum design of the test tanks required that they be large enough to simulate actual fuel tank structure and small enough to be economical to evaluate. The tank size chosen was approximately 9 by 14 by 50 inches. The tank design is a semi-monocoque box beam of 6Al-4V titanium sheet skins and webs, and extruded titanium chords and stiffeners. The end plates and test jig attach fittings are 4130 steel. Reference 2 is the Boeing drawing for the tank details. This type and size of tank is believed to

be the most economical and the least complex construction with which significant and reliable results may be obtained.

2. Two tanks were fabricated and sealed. Tank Number 1 was fillet sealed only. Tank Number 2 was completely faying surface sealed as well as fillet sealed.

3. Tank detail parts were vapor degreased, followed by nitric-hydrofluoric acid etching. Steel parts were descaled in nitric acid, sandblasted, and spray painted with Sermetal W (BMS 14-4) per BAC 5840 for corrosion protection.

4. The tanks were assembled using titanium bolts (BACB30MY6) and collars (BACC30AE6W) from Voi-Shan Manufacturing Company, except for the end plates and access doors where cadmium plated steel bolts were used.

5. In general the sealing was conducted using BAC 5504 procedures as a guide. Dow Corning 77-028 sealant was hand-mixed as described in Section 2.2, Item 2. Some sealant was used immediately after mixing, and some was frozen at -40°F and used within 4 days. The first lot of sealant received (Lot No. 411147, dated 19 November 1969) would not cure properly, even at 300°F. This lot was replaced by Lot No. 1114, manufactured 3 December 1969, which was used for all sealing of both tanks and for the preparation of all Section 3. laboratory test specimens.

6. All structure to be sealed was solvent cleaned with BMS 11-7 cleaner prior to applying primer. Initially some methylethylketone was used instead of BMS 11-7. After solvent cleaning, a thin coat of Dow Corning 77-037 primer (Reference No. E-23-233, dated December 1969) was applied with a gauze pad moistened with primer. The primer was allowed to dry a minimum of 45 minutes at room temperature prior to applying sealant.

7. Freshly mixed (or freshly thawed) sealant was applied using a Semco sealant gun, cartridge and formed nozzle to apply a fillet in one application to the dimensions specified in BAC 5504. Faying surface seals were applied by injecting the sealant on the structure and spreading it out to a thin continuous coating with a spatula or fairing tool.

8. Both access doors were installed with faying surface seals. Tank No. 1 (fillet sealed only) was sealed as shown typically in Figure 1. Block-off faying surface seals were installed (as shown typically in Figure 5) to aid in isolating any fillet leaks which may be evident in the future. Figures 2, 3 and 4 show details of Tank No. 1 during one stage in the assembly and

sealing. The total weight of the sealant fillets in this tank was 2.79 pounds, based on weighing the sealant applied.

9. Tank No. 2 was completely faying surface sealed and fillet sealed. Figure 5 shows details of this tank prior to installation of the spar web.

10. The sealant in each tank received several conditions of cure depending upon location, since the tanks were progressively fabricated and sealed in several steps. Interim cures were generally 1 to 2 hours at 160°F. Upon completion of each tank a minimum cure of 2 hours at 160°F plus 1 hour at 300°F was used. The thermocouples and laboratory test specimens were then installed in each tank (see Figure 6) and the access doors installed using steel bolts in nutplates through faying surface seals. The tanks then received one additional cure of 2 hours at 160°F and 1 hour at 300°F.

11. Completed tanks were leak tested by applying 5 - 10 psi air pressure internally and immersing tanks in water or applying bubble (soap) solution externally. Both tanks exhibited several (up to 9) fastener leaks which were repaired by applying additional sealant over the fasteners on the tank interior. The completed tanks are shown in Figure 6 prior to installation of the access doors.

12. After the sealing of the tanks had been completed, Dow Corning became concerned that sealant lot No. 1114, used to seal both tanks, may not be representative of their best sealant. It was requested that we check the thermal stability of this lot by exposing standard tensile strength and elongation specimens to dry heat at 450°F for 7 days. This was done, along with several other tests, with results as follows:

a. Tensile Strength and Elongation

Exposure (5 Specimens/Condition)	Average % Elongation	Ultimate Tensile Strength, psi Average	Range
(1) Control (No Exposure)	284	680	62
(2) Dry Heat, 450°F, 7 Days	232	503	129
(3) Immersed in Fuel 2 Days 140°F; Plus Dry Heat, 450°F, 7 Days	225	482	160
(4) Same as (3) Except Exposed in a Closed Quart Jar During Dry Heat	220	347	18

b. Reversion resistance was determined by injecting sealant in aluminum and steel tubes 6 to 24 inches long, and with inside diameters of .11 to .25 inch. The sealant generally reverted to an uncured fluid after 3 to 7 days exposure to dry heat at 450°F. The sealant appears unsuitable for enclosed injections.

c. A control peel panel pulled 19 lbs./inch at room temperature with 100 percent cohesive separation.

d. Original application time of this lot, as measured by "snap-time" was 2 - 4 hours at room temperature when the material was first received in December 1969. In January 1970 this had increased to about 48 hours. This indicates a considerable change during storage.

e. The sealant cured adequately in a faying surface after 3 hours at 300°F, or 2 hours at 160°F plus 1 hour at 300°F. Adhesion to the primer was very poor. The sealant exhibited extensive "mud-cracking" or channeling, apparently caused by tearing due to thermal expansion and contraction.

f. Fillet cure properties were as follows:

(1) At room temperature the durometer hardness was only 20 after 10 days. Sealant was tack-free in approximately 48 hours.

(2) At 160°F the sealant was tack-free in 15 minutes and cured to a hardness of 27 in 3-1/2 hours.

(3) At 300°F the sealant cured to a hardness of 37 in 70 minutes.

The sealant appeared to be unsuitable for faying surface or injection sealing. However, the thermal stability appeared satisfactory and it was decided to go ahead and initiate environmental conditioning of the two test tanks.

2.2 Preparation and Testing Of Physical Property Specimens

1. Twenty-one sets of the following specimens were prepared for environmental aging in the two test tanks.

   a. <u>Ultimate Tensile Strength and Elongation per ASTM-D-412</u>
   Four ASTM-D-412 Die C (half-scale: 0.5 by 2.0 in., with 0.125 in. neck) specimens were prepared for each set from slabs prepared as described in paragraph 3, below. Specimens were tested before and after periods of environmental conditioning at a jaw separation rate of 10 inches per minute. Ultimate tensile strength and elongation was reported as the average of four specimens.

   b. <u>Volume Change per ASTM-D-471</u>
   Four specimens approximately 1 by 2 inches were prepared from slabs prepared for each set as described in paragraph 3, below. Volume change, reported as the average of four specimens, was determined after periods of environmental conditioning.

   c. <u>Hardness per ASTM-D-2240</u>
   Type A (Shore A) hardness was determined using the above volume change specimens before and after periods of environmental conditioning. Tests were in accordance with ASTM-D-2240 using a one kilogram weight and a five second reading. The hardness reported was the average of four specimens.

d. <u>Weight Loss</u>

Weight loss was determined using the above volume change specimens. Before and after periods of environmental conditioning, the specimens were conditioned for 24 hours in a dessicator and then weighed immediately. Percentage weight loss was calculated as follows:

$$\frac{(W_1 - W_2) \times 100}{W_1}$$

where W_1 = Weight of sample before aging.

W_2 = Weight of sample after aging.

Percent weight loss was reported as the average of four specimens.

e. <u>Peel Strength</u>

(1) One peel strength panel was prepared for each set using 0.05 by 2.9 by 6 inch panels from 6Al-4V titanium alloy. An equal number of 2.9 by 12 inch strips of 200 mesh stainless steel screen were also prepared. Panels were vapor degreased, followed by nitric-hydrofluoric acid etching. The screen was vapor degreased.

e. Peel Strength (Continued)

(2) Immediately prior to preparing specimens they were solvent cleaned with BMS 11-7 cleaner per BAC 5504. Panels and screen were then primed with a thin coat of Dow Corning 77-037 primer. Primer was allowed to dry a minimum of 45 minutes prior to applying sealant.

(3) Sealant, mixed per paragraph 2 below, was applied to approximately five inches at one end of each panel to a depth of 0.125 ±0.025 inches using a suitable jig. The primed screen was impregnated for five inches on one end and placed on each panel in such a manner that the loose unimpregnated end faced the end of the panel free from sealant. The screen was smoothed down on the sealant carefully to avoid trapping air under the screen. An additional 0.125 ±0.025 inch thick layer of sealant was applied over the unimpregnated screen. The specimens were cured per paragraph 4, below.

(4) Before and after periods of environmental conditioning, two 1.0 inch wide strips were prepared on each panel by cutting completely through the screen and sealant to the metal length-wise along the panel and continuing completely along the unimpregnated

e. Peel Strength (Continued)

(4) (Continued)

screen. The loose end of each 1 inch wide strip in turn was clamped in one jaw of a suitable recording tensile testing machine and the adjacent end of the panel was fastened in the other jaw as shown in Figure 7. Cuts through the sealant under the screen were made so that an initial separation of sealant from the metal panel was promoted. The screen was pulled at an angle of 180 degrees from the panel and at the rate of 2 inches per minute in jaw separation.

(5) Cuts in the sealant to the metal panel at the junction of separation were made at an angle of 45 degrees towards the direction of separation at approximately 0.4 inch increments (approximately every 24 seconds) on the left side of the panel as shown in Figure 7. No cuts were required for 100 percent adhesive failure; however, any cohesive failure was treated as above. On the right side, except for the initial cut to promote separation, cuts were made only as necessary to prevent the sealant from peeling from the screen. All cuts extended completely across the strip being peeled and penetrated completely through the sealant to the panel.

e. <u>Peel</u> <u>Strength</u> (Continued)

(6) The percent cohesive separation was determined from the ratio of cohesive separation area to total cohesive and adhesive separation on both test areas. The cohesive strength was determined during cohesive tear. The average of the cohesive strength, as determined from an extensiometer graph of the right side pull, was recorded. Values recorded during cutting or while load is being picked up after cutting were not included in the average. Panels which were environmentally exposed were tested within 24 hours after removal from the exposure condition.

2. The sealant was mixed in accordance with the following procedure:

a. Weigh onto a clean flat stainless steel plate or pan the correct amounts of base and activator immediately prior to mixing. Do not allow activator to contact the plate.

b. Hand mix by folding and squeezing the sealant compound with a spatula. Mix for a minimum of 5 minutes and until sealant compounds appears uniform.

c. Spread the sealant compound on a clean flat stainless steel plate or pan so that the maximum depth is less than 1/2 inch. De-gas the sealant compound for 10 minutes with a vacuum of 0.24 psia or better.

d. Remove the plunger and plug the nozzle end of a cartridge for the Semco No. 250 gun. Scoop up sealant with a spatula, place it in the open end of the cartridge and drive it down by sharply rapping the nozzle end of the cartridge on something solid. Repeat until the cartridge is filled.

e. De-gas the filled cartridge for five minutes with a vacuum of 0.25 psia or better. A plastic film may be used as an extension of the cartridge to prevent overflow of the sealant. Place the plunger in the cartridge using care to minimize air entrapment.

f. Sealant was used within 2 hours after mixing.

3. Sealant slabs were prepared as follows:

a. Sealant mixed per paragraph 2, was injected into a teflon lined closed mold to a thickness of 0.125 $\pm$0.008 inches.

b. The mold was filled by extruding the sealant from a sealant gun with a Semco No. 440 nozzle. The nozzle was freed of air by a preliminary extrusion of 2 to 3 inches of sealant. During the casting operation, the tip of the nozzle was placed in an injection hole and was not removed until the mold was filled to excess.

c. Sealant was cured 48 hours minimum at room temperature plus 2 hours at 160°F. The sealant slabs were then removed from the mold and cured 1 hour at 300°F.

4. Sealant specimens were cured a minimum of 2 hours at 160°F plus 1 hour at 300°F. Subsequent room temperature cure prior to environmental conditioning was at least 30 days.

5. Ten sets of the above specimens were positioned inside of each test tank (see Tank No. 2 in Figure 6). These specimens were environmentally conditioned along with each tank. Specimens were removed periodically and tested as described above in paragraph 1. One set of specimens was a control (unexposed) set and was held at ambient laboratory conditions. This set was tested with the first set of environmentally conditioned specimens. Test specimens ready for installation in one tank are shown in Figure 8.

6. Physical property specimen test results are reported in Table 1.

2.3 Environmental Conditioning

1. The environmental conditioning of the test tanks was accomplished by placing each tank in separate electrically heated aluminum chambers which were inerted with nitrogen. Figure 9 is a flow chart and Figure 10 is a photograph of the environmental conditioning test equipment. Figure 11 shows one chamber during installation of the side bayonet type strip heaters. Similar heaters were also installed on the bottom of the chambers. The chambers were insulated on the exterior with firebrick covered with fiberglas batting.

2. Thermocouples were placed on three heaters (two side and one bottom) for each chamber. These six thermocouple temperatures were continuously recorded on one six-point recorder. This recorder was equipped with an over-temperature control to preclude over-heating of the heaters.

3. Thermocouples were installed in the tank interiors in the locations indicated in Figure 6. Thermocouples were also inserted in drilled holes in one exterior corner of each heavy steel end plate at the ends of each tank. Three thermocouple temperatures for each tank (top plate, tank interior, and bottom plate) were continuously recorded on the second six-point recorder. This recorder was also equipped with an over-temperature control to preclude over-heating of the tanks. For each chamber, the side and bottom heater temperatures were separately controlled by two Thermac Temperature Controller and Power Regulator Units (one for side heaters, one for bottom heaters). Each side heater Thermac Unit controlled side heater temperatures by means of a thermocouple in each tank interior. Each bottom heater Thermac Unit controlled bottom heater temperatures by means of a thermocouple in each tank bottom plate. A

4. Initial heat up of one environmental chamber was conducted with an old previously tested titanium tank installed in the chamber to check tank temperature variations. This chamber, in its original configuration, had side heaters only. Temperature variations in any horizontal plane around the test tank were less than 10°F which was considered satisfactory. However, the temperature variations in a vertical direction was over 100°F (tank bottom was only 200°F when the tank top was 312°F). It was therefore considered necessary to install heaters on the bottoms of the chambers.

5. Initially it had been planned to expose the tanks to liquid fuel for 1 hour at 140 - 150°F each working day (5 times a week). However, excessively long times were required to heat up the tanks for the elevated temperature fuel vapor exposure (approximately 2 hours) and to stabilize at the desired temperature range (approximately 8 hours, subsequently reduced to about 3 hours). It was therefore agreed that one weekly liquid fuel exposure of approximately 16 hours is satisfactory. This will allow a weekly elevated temperature fuel vapor exposure of approximately 130 hours. A

6. During the elevated temperature environmental conditioning nitrogen and liquid fuel were added to each tank at the following rates:

   a. Nitrogen was added at a rate of one tank volume every three hours. With a tank volume of approximately 27 gallons, this was a nitrogen purge rate of approximately 570 cubic centimeters per minute.

   b. Liquid fuel was added at a rate of 0.1 percent tank volume every 90 minutes. This was calculated to be approximately 1.5 cubic centimeters per minute.

   c. Nitrogen and fuel additions were metered using shielded type compact flowmeters (size 12 and 11 respectively) from Roger Tilmont Industries, Incorporated.

7. Fuel used for the first eight environmental conditioning cycles was MIL-J-5161 Grade II with 18.2 percent aromatics. This fuel was purchased from Crystal Refining Company, Carson City, Michigan. Subsequently all fuel was ASTM D1455 Jet A with 14.8 to 15.3 percent aromatics, and purchased from Standard Oil of California, Salt Lake City, Utah. A

8. The environmental conditioning for each cycle is shown in Tables 2 and 3 for Tank Numbers 1 and 2 respectively.

2.4 Dynamic Load Cycling

2.4.1 Description Of Dynamic Test Device

1. The Dynamic Test Device was originally developed and used under Boeing-Air Force Materials Laboratory research contract reported in Reference 1. An improved version of this test device (Figure 12) was subsequently built under a Boeing research program. Torsional loads are applied by mounting a test tank horizontally as a cantilever beam, with one end firmly attached to the steel frame and the other end subjected to dynamic loading using a hydraulic ram. Cyclic loading is accomplished by a timer which operates a valve causing the ram to cycle.

2. To evaluate a tank at elevated temperatures, an insulated chamber attaches to the test jig, completely enclosing the test tank. The chamber is a stainless steel cylinder, consisting of two half-sections with fiberglass insulation, as shown in Figure 13. Heating of the tank is accomplished by blowing hot air between the tank and the insulated chamber.

3. To evaluate a tank at low temperatures, cold JP-4 fuel is circulated through the tank. The fuel is cooled to the desired temperature by pumping it through a coil immersed in a mixture of trichloroethylene and dry ice (see Figure 12).

2.4.2 Determination Of Torsional Load Levels

1. Commercial Airplane Group stress analysis on the test tanks indicated that they were shear and fatigue resistant up to 159,000 inch-pounds torsional loading. Some tests were conducted to determine the range of joint deflections that would be obtained during cyclic loading. The tank used for this purpose was one used previously in an Air Force Material Laboratory Contract. This tank had been dissembled, stripped, cleaned up, and reassembled. This tank is identical to the two tanks being tested in this program, with the following exceptions:

   a. It had been used previously at torsional loads up to 270,000 inch-pounds.
   b. It was assembled with 0.25 inch steel nuts and bolts, torqued to 60-100 inch-pounds.
   c. The access door is 0.10 inch thick 2024-T3 aluminum with two reinforced 4 inch diameter holes to permit measuring joint deflections by hand.
   d. The center rib web contained no cut-outs.

2.4.2 (Continued)

2. It was very difficult to measure the joint gaps due to lack of visibility and the restricted access through the holes in the access doors. Joint gaps were measured manually by inserting small feeler gauges in the joints. Many of the extrusions used in the tank had curved edges making exact measurement impossible. The smallest gauge that could be handled under these circumstances was 3 mils. Initially (prior to loading) the joint gaps were generally 0 - 3 mils, with the following exceptions:

   a. Center rib attachments to the spar web and the upper and lower skins: 3 - 13* mil gaps.

   b. Spar web to spar chord adjacent to lower skin: 3 - 11* mil gaps.

   c. Lower skin at longitudinal stiffener: 13 - >18* mil gaps.

   d. Upper skin at longitudinal stiffener: 3 - 5 mil gaps.

*Larger joint gaps were due to, (1) dimensional variations in titanium extrusions purchased back in 1963, (2) poor fit of center rib, and (3) possibly some permanent deformation of skins and web when previously used.

2.4.2 (Continued)

3. Torsional loading of this tank at 160,000 inch-pounds produced little measurable increases in joint gaps. Two places on the spar web increased--one 2 mils and one 4 mils. Torsional loading at 200,000 inch-pounds produced several increases in joint gaps:

   a. Spar Web: 1 place 4 mil increase.

   b. Upper Skin at Longitudinal Stiffener: 1 place 2 mil increase.
      1 place 5 mil increase.

   c. Lower Skin at Longitudinal Stiffener: Several places where gaps increased 4 - 10 mils.

4. Loading at this level did not appear to produce permanent visible deformation of the structure. However, when loaded to 240,000 inch-pounds torque, some slight permanent deformation of lower skin was observed. Therefore, the load levels chosen for the two test tanks was 160,000 inch-pounds (many cycles) and 200,000 inch-pounds (fewer cycles).

2.4.3 General Test Procedure For Dynamic Cycling and Leak Testing Of Tanks

1. Environmental conditioning of each test tank per Section 2.3 was interrupted periodically to conduct cyclic torsional load testing at a high temperature, low temperature, and at room temperature.

   Torsional loading was conducted as follows:

Tank Test Temperature	500 Torsional Load Cycles At	10 Torsional Load Cycles At
Room Temperature	160,000 inch-pounds	200,000 inch-pounds
Low Temperature (-45 to -50°F)	160,000 inch-pounds	200,000 inch-pounds
High Temperature (426 to 441°F)	128,000 inch-pounds	160,000 inch-pounds

   Load cycling rate was generally 8 - 12 cycles per minute. Each cycle included torsional loading in both directions.

2. After dynamic cycling at each temperature, each tank was leak tested by applying 10 psig pressure internally, applying aqueous soap solution to the tank externally, and recording all leakage as indicated by soap bubbles at the external leak location. Leakage is recorded in Appendix A.

2.5 Evaluation Of Tanks After Four Cycles Of Environmental Conditioning

A

1. The primary test areas of each tank were considered to be the upper and lower skins and the spar. These areas, including the center rib, more closely simulated typical integral fuel tank structure and therefore the performance of the sealant in these areas is of primary significance. Areas of the tank that are out of the primary test area are the tank ends and the access door. The ends of the tank are composed of heavy steel end plates and jig attach fittings. These areas do not contain typical structure and may be subjected to unrealistic loads and deflections. The access door was faying surface sealed and attached by a single row of removable steel bolts. Door joint deflections and faying seal width is not typical of fuel tank structure. Therefore the tank ends (including areas under the jig attach fitting fingers), and the access door are not part of the primary test areas, and the performance of the sealant in these areas is of secondary importance only. These areas are designated secondary test areas.

2.5 (Continued)

2. Upon completion of four cycles of environmental conditioning (524 hours exposure to fuel vapor at 415-441°F, per Tables 2 and 3) both tanks were removed from the environmental chambers and leak tested. Leakage is summarized in Table 4. Specific leak locations are shown in Appendix A. There was considerable leakage in the secondary test areas (tank ends and access door). The reasons for these leaks appeared to be due to, (1) extrusion of sealant from the injections, and (2) loss of adhesion of the sealant to the Sermetal W painted steel parts in these areas. Considerable work was expended to clean up these leaking areas on the exterior of the tanks and to seal these leaks externally with the test sealant. Since the tanks were not opened at this point the cause of the fastener leaks in the primary test areas was not evident.

3. Both tanks were installed (separately) on the Dynamic Test Device and load cycled per Section 2.4.3 at an elevated temperature of 428-439°F. Prior to heating, one liter of test fuel was added to Tank No. 1, and three liters were added to Tank No. 2. The tanks were continuously purged with nitrogen and were not pressurized.

   On completion of dynamic cycling, the tanks were again leak tested. Leakage is shown in Table 4.

2.3 (Continued)

4. The tank access doors were removed, and examination of the interior revealed the following:

   a. Fastener Leaks

      The fastener leaks were generally due to, (1) loss of sealant adhesion to titanium adjacent to fasteners (see Figure 14), or (2) small cracks in the sealant. Numerous fasteners not leaking also exhibited these conditions. The causes of the 22 fasteners leaking in Tank No. 1 were generally much less evident than for Tank No. 2.

   b. Joint Leaks

      Although only Tank No. 2 contained joint leakage, both tanks exhibited cracking and/or loss of adhesion of joint fillets at corners (where fillets make a 90 degree turn). At the center rib Tank No. 1 and No. 2 had two and five cracked corner fillets respectively (see Figures 15, 16, and 17). Both tanks also had cracked and loose fillets at the corners of the end plates.

2.5 (Continued)

5. Apparent causes of sealant failures were:

   a. Poor sealant adhesion to titanium.

   b. Low adhesive and cohesive strength of sealant at elevated temperatures (peel strength is only 2.5 pounds per inch at room temperature per Section 2.2).

   c. Thermal expansion problems (expansion of injection seals under fillets in corners may be the cause of corner fillet failures).

   d. Undersize applications of sealant over fasteners, or entrapped air bubbles in sealant may have caused a few fastener leaks.

6. Joint fillets not in corners were generally satisfactory in both tanks. The fifteen joint leaks in Tank No. 2 were generally adjacent to leaking fasteners or corners where joint fillets were cracked or loose. Tank No. 2 was completely faying surface sealed, but exhibited 15 joint leaks and 51 fastener leaks. Tank No. 1, faying sealed at center rib only, exhibited no joint leaks and only 22 fastener leaks. This was a paradox not readily understood. It appeared that the faying surface seals in Tank No. 2 were completely ineffective.

7. It was decided at this point to, (1) repair discrepant sealant in Tank No. 1 and continue testing, and (2) to terminate testing of Tank No. 2 because of excessive leakage.

2.6 Repairs and Continuation Of Testing Of Tank No. 1

2.6.1 Repairs Of Tank

1. Four pounds of Dow-Corning 77-028 sealant, Lot No. 104105 and one pint of 77-037 primer, Lot No. E23-276, were received on April 24, 1970, to repair Tank No. 1. Preliminary testing showed that the new sealant would not adhere to cured unexposed sealant, and when applied to sealant previously exposed to fuel the new sealant would not cure at the interface. Subsequent testing showed that if the fuel exposed sealant was dried to remove the fuel (overnight at about 425°F) and the exposed surface then cut away and fresh sealant applied, marginal compatibility (2 pounds per inch peel strength) could be obtained. The tank was therefore exposed to dry heat for 18 hours at 425°F. Some additional small cracks in fillets and areas of poor adhesion were discovered during repairs. The sealant was very easy to damage. Cutting away discrepant sealant frequently caused adjacent sealant to crack or come loose.

2.6.1 (Continued)

2. All fillets showing cracks and/or poor adhesion were removed and replaced with new sealant. Cleaning of the metal was accomplished with aluminum wool, Scotchbrite, and gauze dampened with BMS 11-7 cleaner. Leakage before and after repairs is summarized in Table 5 (Leakage Test No. 2 and 3). In general, the repairs eliminated very few leaks. Sixteen old fastener leaks were eliminated, but 20 new ones developed. Additional leakage was probably due to, (1) damage of sealant during repairs, and (2) to the need to bake the aged sealant to remove the fuel in order to make the aged sealant repairable. Leakage at the tank ends was not reduced appreciably, but load cycling and additional environmental conditioning was still considered feasible.

2.6.2 Dynamic Load Cycling After Repairs

1. The tank was installed on the dynamic test device and load cycled per Section 2.4.3 at -47 to -49°F and at room temperature. A detailed leak test could not be conducted readily at this point since the tank was still on the dynamic test device and was wet with fuel which prevented the soap solution from working properly. However, leakage recorded is shown in Table 5 (Leakage Test No. 4).

2.6.2 (Continued)

2. The tank was subsequently heated to 429-441°F and load cycled for 20 cycles at 160,000 inch-pounds torque plus 30 cycles at 128,000 inch-pounds. This hot load cycling was conducted to see if the new fillet repairs would crack with no prior environmental conditioning. Leakage is summarized in Table 5 (Leakage Test No. 5). Ten joint leaks and 80 fastener leaks were present in the primary test areas. Approximately 54 of the fastener leaks were new. Seven of the ten joint leaks were adjacent to leaking fasteners. These fasteners were probably the source of seven joint leaks, and may be the source of the other three.

3. Examination of the tank interior after the above described load cycling can be summarized as follows:
   a. The new fillets installed during repairs showed no major cracking, but there were numerous areas of loss of adhesion to the structure and the old sealant. This was about 20 percent of the repaired areas. There were some small cracks in new and old sealant in fillets at ends of tank in corners.
   b. Old sealant fillets in tank ends show additional loss of adhesion.
   c. Cause of the many additional fastener leaks was not apparent.
   d. The fillets in general continue to look good.

2.6.2 (Continued)

4. After the above load cycling the access door was again leaking badly. During the room temperature cycling it was observed that the access door was moving (sliding) excessively at the faying surface due to the oversize fastener holes in the door (which were necessary to make the door fit). A new door was fabricated and installed prior to continuation of environmental conditioning per Section 2.6.3. A small amount of a low expansion sealant (Dow Corning 77-066, Lot Z-40-15) was used in the prepack channels at each end of the access door; however, this was not considered to be a good evaluation of this material.

2.6.3 Evaluation Of Tank No. 1 After 12 Cycles Of Environmental Conditioning

1. Tank No. 1 was exposed to eight additional cycles of environmental conditioning per Table 2 (Cycles 5 through 12). Accumulated exposures after 12 cycles were as follows:

   a. Liquid Fuel Exposure

Ambient	-	139°F	204.6 Hours
140°F	-	150°F	173.0 Hours
152°F	-	175°F	30.5 Hours
Total Liquid Fuel Exposure			408.1 Hours At Ambient To 175°F

2.6.3 (Continued)

b. Fuel Vapor Exposure

Ambient	-	400°F	118.0 Hours
401°F	-	425°F	51.3 Hours
415°F	-	441°F	624.5 Hours
426°F	-	441°F	939.1 Hours
443°F	-	457°F	25.2 Hours
Total Fuel Vapor Exposure			1758.1 Hours At Ambient To 457°F
			1563.6 Hours At 415 - 441°F

2. The tank was then removed from environmental conditioning and leak tested. Leakage is shown in Table 5 (Leakage Test No. 6).

3. The tank was then load cycled per Section 2.4.3 at all three temperatures and again leak tested (see Table 5, Leakage Test No. 7). There are 24 joint lengths, separated from each other by faying surface seals, in the primary test areas (eight each on top skin, lower skin, and spar). It is difficult to summarize the leakage since many leaks "come and go" (as seen by comparing the total leaks obtained during Leakage Test No. 6 with those obtained during (Leakage Test No. 7).

2.6.3 (Continued)

4. The tank access door was removed and the sealant examined. Appendix A (Page A-11) shows the locations and types of joint fillet defects found, which can be summarized as follows:

Symbol	Type Of Defect	Number Of Defects
15	Small crack or loss of adhesion. Crack generally penetrates to substrate with some accompanying loss of adhesion.	40
14	Crack in repair fillet.	1
13	Loss of adhesion of repair fillet to titanium.	2
12	Loss of adhesion of repair fillet to original fillet.	2

2.6.3 (Continued)

5. In an effort to determine where the leaks originated, an interior leakage analysis test was conducted by placing a transparent access door on the tank, applying 5 psig negative (vacuum) pressure to the tank interior and observing leaks on the interior bubbling through a 1 to 2 inch layer of water. It was desired to determine the following:

   (1) Whether the exterior leaks were primarily caused by leaking fasteners or joint fillets.

   (2) Whether joint fillets were leaking in straight fillet areas or only at corners (where cracks and loss of adhesion had made previous repairs necessary).

   Interior leakage obtained by this method is shown in Appendix A (Page A-12) and the numbers of leaks obtained can be summarized as follows:

Origin Of Leak	Relative Rate Of Leakage		
	One Bubble Indication (1)	Small Continuous Bubbling	Large Vigorous Bubbling
Sealant over fastener	41 Leaks	9 Leaks	1 Leak
Joint fillet at or near corner	8 Leaks	4 Leaks	2 Leaks
Joint fillet in straight-seam areas	28 Leaks	7 Leaks	0 Leaks
Uncertain (Joint fillet and sealant over fastener are continuous)	0 Leaks	3 Leaks	0 Leaks

(1) One bubble forms on sealant but does not break and reform. These can be classified as possible or suspected leaks only.

2.5.3 (Continued)

6. An attempt was made to correlate the exterior leakage (Table 5), the interior sealant defects (Item 4 above) and the interior leakage (Item 5 above). In general, there is very little correlation; however, the following items are considered significant:

   a. On the tank exterior 11 to 16 percent of the fasteners and 25 to 33 percent of the 24 joint areas are leaking.

   b. On the tank interior 10 fastener leaks were confirmed, with 41 more suspected. Thirteen joint fillet leaks were confirmed, with 36 more suspected.

   c. Of the 13 confirmed joint fillet leaks, 7 leaks were in straight seam areas (not at or near corners). None of these 7 leaks were at the 32 defects mentioned in Item d., below.

   d. Forty defects (small cracks and/or spots showing loss of adhesion) were observed in the joint fillets. Thirty-two of these defects were in straight seam areas (not at or near corners).

7. At this point it appeared that the sealant was cracking and losing adhesion because of the low physical properties resulting from inadequate resistance to the environmental conditioning and structural loading. Continuation of the testing of this tank in Section 2.7 was primarily to evaluate sealant repair procedures.

2.7 Evaluation Of Sealant Repair Procedures In Tank No. 1 A

1. Defective and/or leaking joint fillets were removed from 14 locations (crosshatched areas in Appendix A, Page A-12). In order to evaluate the repairability of the fuel aged discrepant sealant, these areas were repaired in August 1970 using sealant received from Commercial Airplane Group. The following repair procedure was used:

   a. Fuel aged sealant was dried 16 hours at 420°F to remove fuel.

   b. Dried sealant was then scrubbed with cheesecloth and BMS 11-7 solvent to clean and remove gloss.

   c. Structure was cleaned with Scotchbrite and cheesecloth wet with BMS 11-7.

   d. A thin coat of primer was applied to structure only and dried 1 to 2 hours.

   e. A thin brush coat of freshly thawed sealant was applied to structure and old sealant with acid brushes.

   f. Sealant fillets were applied within one hour.

   g. Sealant was cured overnight at room temperature, 2 hours at 160°F and 1 hour at 300°F.

2.7 (Continued)

2. It was not possible to clean the old sealant without causing some loss of adhesion. No attempt was made to repair the leaking fasteners. The ends of the tank (out of the primary test areas) were still leaking considerably and not considered repairable. (Fuel leakage during the 16 hour liquid fuel exposure portions of environmental conditioning cycles 8 through 12 ranged from 1200 to 10,300 milliliters.)

3. The tank was exposed to 8 additional cycles of environmental conditioning per Table 2 (Cycles 13 through 20). Total accumulated exposures after 20 cycles were as follows:

a. Liquid Fuel Exposure

Ambient	-	140°F	236.6 Hours
140°F	-	150°F	290.5 Hours
152°F	-	175°F	54.0 Hours
Total Liquid Fuel Exposure			581.1 Hours At Ambient To 175°F

b. Fuel Vapor Exposure

Ambient	-	400°F	220.6 Hours
401°F	-	425°F	67.1 Hours
415°F	-	441°F	637.0 Hours
426°F	-	441°F	1983.1 Hours
443°F	-	457°F	42.2 Hours
Total Fuel Vapor Exposure			2950.0 Hours At Ambient To 457°F 2620.1 Hours At 415°F To 441°F

The fuel vapor exposure after the repairs reported in Item 1, above was 1056.5 hours at 415°F to 441°F.

2.7 (Continued)

4. The tank was removed from environmental conditioning and leak checked by putting approximately 2 inches of water in the tank, installing an acrylic door, applying 5 psi of vacuum and observing bubbles. Results are summarized in Appendix A (Page A-13). There were 2 leaks in one of the 6 repaired areas of the upper panel. In the lower panel there was 1 leak in one of the 3 repaired areas, one new fillet leak and 4 new fastener leaks. The spar had 2 of 5 repaired areas leaking and 1 new fastener leak.

   A number of cracks in the thin faired portion of the fillets was observed. Under these cracks are bare spots where the sealant separated adhesively. A portion of the cracked fillets were removed in 4 locations. Also, one leaking repair in the spar was removed, revealing a section where there was no adhesion.

   These were all repaired according to the procedure described in Item 1 above, except that 400 grit aluminum oxide sandpaper was used to abrade the surface. Following repair and an overnight bake in air at 180°F, three of the repairs were removed to check adhesion. Good adhesion had been maintained. These three repairs were then repaired again.

2.7 (Continued) A

5. The tank was closed up and placed in the environmental chamber where it was filled with fuel and exposed at room temperature for 14 days. It was refilled periodically as fuel leaked out.

6. The tank was then placed on the dynamic test device and load cycled as follows:

   a. 500 Cycles at 426 to 441°F and 128,000 inch-pounds torque.
   b. 510 cycles at room temperature (500 cycles at 160,000 inch-pounds torque and 10 cycles at 200,000 inch-pounds torque).

7. The tank was then environmentally conditioned in accordance with Table 2, Cycles 21 through 31. Environmental conditioning was the same as used previously (Section 2.3) except the liquid exposure for each cycle was reduced to one hour due to the excessive leakage of this tank.

8. Environmental conditioning of Tank No. 1 was terminated upon completion of Cycle 31 due to program cancellation in March 1971. In August 1971 the program was reactivated and Tank No. 1 was leak tested and examined after 31 environmental conditioning cycles (see Table 2). The leakage observed is shown in Appendix A (page A-13). With the exception of the new leak at the 1/4" crack in the spar fillet, B

all new leaks were small and nearly undetectable. Only two of these new leaks were in any of the repairs made after 20 environmental conditioning cycles and they were in the same repaired section. There were 10 new leaks in repairs made after 12 environmental conditioning cycles, and 12 new leaks in original sealant. Note however, that no new leaks occurred in seven of the fourteen repairs made after 12 environmental conditioning cycles. These repairs had accumulated a total of 2298 hours fuel vapor exposure, while the repairs after 20 environmental conditioning cycles had accumulated 1242 hours fuel vapor exposure. The only difference in the repair procedures was that in the latter case the surfaces to be repaired were abraded using 400 grit emery paper rather than the Scotchbrite used in the former case. The test results do not indicate that the severe abrading was any more effective.

9. Testing of Tank No. 1 is considered complete. No additional testing of this tank is planned.

3. TESTING OF TANK NO. 3

3.1 General Description

1. This tank was designed, fabricated, and sealed by Commercial Airplane Group per Reference 4, and was intended to simulate the SST fuel tank structure more closely than the first two tanks. The tank was built to much closer tolerances than was possible for the first two tanks (which were built in 1934 with badly warped extrusions that could not be properly machined). The tank is 10 by 18 inches in cross section and 50 inches long. Skin and spar webs are chemically milled and the top skin contains a full size elliptical shaped fuel tank access door. The tank is of conventional skin and stiffener type construction with joggled internal skin stiffeners, and exterior skin and spar stiffeners. It has no intermediate ribs. The tank was primarily fillet sealed with Dow Corning 77-028 (same sealant used in previous tanks), but also contained a few primary faying surface and injection seals on the lower skin.

3.2 Initial Preparation and Testing Of Tank

1. Tank No. 3 was leak tested initially by applying 5 psig internal pressure and immersing the tank in water. One fastener was leaking (see Appendix B, Page B-1).

2. Holes in the tank end plates (for installation on the dynamic test device) were located, drilled and tapped (sixteen 1/2 inch diameter holes in each end, drilled about half way through the 2 inch thick end plates).

3. Six thermocouples were installed in the tank (two each on upper and lower skin stiffeners, and one in the center of each end plate).

4. The tank was placed on the dynamic test device and initial torsional loads were applied to measure the extent and depth of web shear wrinkling at five locations. Test data is shown in Appendix C. Initial web buckling was obtained at approximately 50,000 inch-pounds of torque. Results showed that at 216,000 inch-pounds of torsion (typical flight load), significant web wrinkles do extend into the pad-up areas of the chem-milled webs and even into the chords and stiffeners.

▼ A

3.3 Dynamic Load Cycling

3.3.1 General Test Procedure

Dynamic load cycling of Tank No. 3 was conducted per Section 2.4.3 with the following exceptions:

a. Torsional load levels were as follows:

Tank Test Temperature	500 Torsional Load Cycles At
Room Temperature	216,000 inch-pounds
Low Temperature (-45 to -50°F)	216,000 inch-pounds
High Temperature (426 to 441°F)	172,800 inch-pounds

b. Leak testing was conducted at 5 psig with the tank immersed in water.

3.3.2 Initial Load Cycling Prior To Any Environmental Conditioning

1. The tank was load cycled per Section 3.3.1 at room temperature, at the high temperature, and at the low temperature. The low temperature cycling was terminated at 330 cycles when the ends began leaking fuel and it was discovered that the bolts in the end plates were loose. Both rows of bolts in the upper skin and both spars were very loose (2 to 3 turns). Only a few of the bolts in the lower skin were loose and these only slightly. Four bolts in the upper skin had broken at the threads. All bolts were subsequently replaced with CRES bolts, B30LM3HU, lock-wired in place. B

3.3.2 (Continued)

2. After the above load cycling (but prior to installing new bolts) the tank was leak tested. Leakage is shown in Appendix B, Page B-2. There were two joint leaks in the lower skin and two corners were leaking at the ram end. The fastener leak was the same one leaking prior to any load cycling. Based on the fuel leakage from the corners during dynamic cycling it is probable that more than two corners would be leaking if the tank could be leak tested under load.

3. The tank was then opened and the interior examined. Numerous cracks in the fillets were found in the lower skin as shown in Appendix B, Page B-3. Eight cracks were in joint fillets and two were in sealant over fasteners in the primary test areas of the tank (excluding tank ends). Cracks were also found in four of the eight corners of the tank (four of these cracks appeared to be due to separation of repair sealant). Vacuum leak testing at 3 psig with a layer of water C on the bottom skin revealed the source of two exterior leaks to be fillet seal cracks (see Appendix B, Page B-3). Portions of the fillets containing cracks at the two leak locations were carefully removed to preserve the cracked section. Adhesion was good except at one fastener.

3.3.2 (Continued)

4. The tank was then mounted on the dynamic test device. Members of the Stress Group were in attendance to observe the wrinkles formed during loading and to assess the effect on structural life of the tank. It was concluded that the tank would easily last for the program life. The wrinkles were very prominent. Three to 4 thousandths of an inch gap could be measured between the skin and stiffeners on the outside of the tank in one or two places; however, deflections inside were less than .0015 inch (in the areas where there were no fillets and measurement was therefore possible). B

5. The tank was load cycled at room temperature for an additional 1500 cycles in an effort to see if additional fillet cracks would be obtained or if existing cracks would grow.

6. The two areas where cracked fillets were removed (Item 3) were repaired, and the tank was load cycled per Section 3.3.1 at the elevated temperature (426 to 441°F). Small test specimens were attached to the tank during the loading. They consisted of heavy fillets on 6Al4V titanium. One was for control, one had been "puddled" extensively, and one was permanently deformed to put the fillet in tension. Following the exposure with the tank, these specimens were thermally shocked a number of times by heating to 440°F and immediately placing in contact with a -40°F surface. In no instance did we produce new cracks or extend the old ones.

3.3.2 (Continued)

7. The tank was then leak tested (see Appendix B, Page B-4). One new leak was found on the lower skin. The tank was opened and the fillets examined (see Appendix B, Page B-5). Five small fillet cracks were found on one spar fillet. These were very small and <u>may</u> have been overlooked previously. Three small cracks were found on fillets of the lower skin (1 seam fillet crack and 2 fastener fillet cracks).

8. Leaks were repaired in four areas of the tank identified by B in Appendix B, Page B-6. These repairs stopped all but one leak (which had not been repaired because the interior source could not be identified).

9. The length of all existing cracks was measured and recorded (see Appendix B, Page B-3).

10. Physical property test specimens were installed in the tank, the original access door installed, and environmental conditioning was initiated on January 13, 1971.

A

3.4 Preparation and Testing Of Physical Property Specimens For Tank No. 3

1. Physical property test specimens were prepared from slabs of cured sealant provided by Commercial Airplane Group. Slabs were cast from Batch No. 205180 on October 8, 1970. Ten sets of specimens were prepared. Each set contained:

   a. 15 Tensile Strength and Elongation Specimens.

   b. 3 Volume Change Specimens.

   Five peel panels (prepared by Commercial Airplane Group) and nine sets of the above specimens were placed in Tank No. 3. One set was a Control (Unexposed) Set.

2. In general, test specimens were prepared and tested in accordance with Section 2.2. Tensile Strength and Elongation Specimens were tested at three temperatures (5 specimens per condition): 75 ±5°F, 450 ±5°F and -50 ±5°F. B

3. Test specimens were removed from the tank and tested per Section 2.2, paragraph 1. Results obtained are reported in Table 6. B B

3.5 Environmental Conditioning Of Tank No. 3

1. Environmental conditioning of Tank No. 3 was generally accomplished as described in Section 2.3. The specific environments for each environmental conditioning cycle is shown in Table 7. Eight cycles were completed when this program was cancelled in March 1971. The tank remained closed up and essentially empty (in the Figure 10 Environmental Chamber) until work resumed in August 1971.

2. The tank was removed from the environmental chamber and leak tested at 5 psi while immersed in water. Leakage is shown in Appendix B, page B-7. Six of eight corners were leaking badly. Five small seam leaks were found on the lower skin. Examination of the interior revealed numerous new cracks. The six leaking corners contained large cracks. When the tank was dried out (at 310°F for 16 hours) in preparation for repairs, additional cracks appeared and many increased in length. Locations of interior cracks are shown in Appendix B, page B-8. A 3 psi vacuum leak test with water on the interior lower skin revealed 7 interior leaks as shown in Appendix B, page B-9. These leaks were small. The numerous cracks in the sealant fillets were the main item of concern. Those in the corners were gapping and up to 1.5 inches long. New cracks were found in fillets at ends of lower skin stiffeners. It appeared that the thicker the fillet the greater the chances of it cracking. Eight

B

cycles of environmental conditioning did not cause the fillet cracks found initially (prior to environmental conditioning) to grow or cause leaks.

3. The 6 leaking corners were repaired and the tank was again leak tested. See Appendix B, page B-10. Two of the six corners were still leaking. One new small seam leak was found in the upper skin.

4. The tank was installed on the dynamic test device and load cycled at all three temperatures per Section 3.3.1. The tank was removed from the dynamic test device and leak tested. Leakage is shown in Appendix B, page B-11. Seven new leaks were observed, but were not large enough to require repairing. Examination of the tank interior showed no significant changes.

5. Environmental conditioning of Tank No. 3 was resumed on September 7 with Cycle No. 9, and was terminated on December 23, 1971 with Cycle No. 22. See Table 7.

3.6 Dynamic Load Cycling of Tank No. 3 After 22 Cycles of Environmental Conditioning

C

1. After 22 cycles of environmental conditioning (see Table 7) Tank No. 3 was leak tested by immersion in water as previously described. Leakage is shown in Appendix B, page B-12. There were 9 new leaks, including 2 new corner leaks. One of the 2 new corner leaks was a previously repaired corner, the other was a leak in the original sealant. Except for the corners all leaks were very small. The access door was leaking badly also.
2. The tank was opened and the interior examined. The leakage of the access door was due to 100 percent compression set of the fluorosilicone door seal. All defects in the tank interior are recorded in Appendix B, page B-13. There were approximately 28 new defects in the tank since the last examination (after 8 environmental conditioning cycles, page B-8).
3. The tank was again placed on the dynamic test device and load cycled at all three temperatures per Section 3.3.1. The tank was removed and leak tested immersed in water. Leakage is shown in Appendix B, page B-14. Six new leaks were obtained, for a total of approximately 24 leaks. A few leaks traveled to adjacent areas or disappeared. All leaks, except for the 6 leaking corners, were very small.

4. The six leaking corners were repaired and the tank leak tested. Leakage after repairs is shown in Appendix B, page B-15. While leak testing, an additional corner (F-2) was found to be leaking badly. This appeared to be due to a crack approximately 1/4 inch long in the sealant that apparently opened up when the tank was dried out prior to repairs. The sealant was chamfered back and the crack well filled with fresh sealant.

5. Table 8 shows the corner leakage history of tank No. 3.

6. The door was installed using a gasket made of fluorosilicone rubber sheet. The tank was placed in the environmental chamber and environmental conditioning was resumed on 23 February 1972.

3.7 Dynamic Load Cycling of Tank No. 3 After 39 Environmental Conditioning Cycles (August 1972)

1. After completion of 39 environmental conditioning cycles (see Table 7) Tank No. 3 was removed from the environmental chamber and leak tested. Leakage is shown in Appendix B, page B-16. Prior to repairing leaks the tank was dried out 16 hours at 400°F. The tank interior was then examined and fillet cracks are shown in Appendix B, page B-17. A few new cracks were evident. It was subsequently decided to repair leaks <u>after</u> load cycling the tank since no primer was available at this point.

2. The tank was placed on the dynamic test device and load cycled at all three temperatures per Section 3.3.1. The tank was removed and again leak tested. Leakage, shown in Appendix B, page B-18, can be summarized as follows as compared with leakage after 22 environmental conditioning cycles:

Leak Location	After 22 Cycles	After 39 Cycles
Corners (see Table 8)	4	4
Seams	15	7 (1)
Fasteners	3	0

(1) 4 new seam leaks; 12 seam leaks disappeared.

There was no significant increase in interior fillet cracks after the load cycling (see page B-17).

3. Of the four leaking corners only two (R-1 and R-2) exhibited interior defects which appeared to be the cause of the leakage. These two corners were leaking significantly and were therefore repaired as follows:

   a. All defective sealant in corner area was removed, including sealant in the two injections adjacent to each corner.

   b. Metal was scrubbed with aluminum oxide paper and cleaned with BMS 11-7 cleaner.

   c. A thin coat of a new two-part primer (Dow Corning 77-123) was applied to the metal. This primer replaced the previously used Dow Corning 77-037 which is no longer available.

   d. The primer was dried 90 minutes at room temperature, and freshly mixed 77-028 sealant applied. Sealant was cured immediately at 300°F for 1 hour minimum.

   The two repaired corners were leak tested and showed no leakage.

4. Test specimens removed from the tank were tested as before and results are reported in Table 6.

5. Remaining test specimens and new thermocouples were installed in the tank and environmental conditioning was resumed on 6 September 1972. The fluorosilicone rubber gasket on the door was in good condition and was not replaced.

3.8 Repair of Tank No. 3 After 46 Environmental Conditioning Cycles (November 1972)

1. After completion of the liquid fuel exposure portion of environmental conditioning cycle no. 47, approximately 11.5 gallons of fuel had leaked out of the tank into the environmental chamber. Prior leakage had been gradually increasing from 3/4 gallon (cycle no. 42) to 5-1/2 gallons (cycles 45 and 46). Due to the excessive leakage the tank was removed from the environmental chamber and leak tested with 5 psig air pressure. All 8 corners of the tank were leaking (see Appendix B, page B-19).
2. Since most of the leakage would be from those corners that were at the bottom of the tank during environmental conditioning, it was decided to repair only these four corners (R-1, R-2, R-3, and R-4). Corners R-1 and R-2 were previously repaired in September 1972 (see Table 8), and it was apparent that the new sealant in the injections adjacent to these corners had expanded and failed the fillets in these corners. These two corners were repaired with new fillets and the adjacent injections were cleaned out and left empty. All 4 corners were repaired using the procedure used in September (see Section 3.7, Item 3) except sealant was cured overnight at ambient temperatures prior to curing at 300°F.
3. After the repairs the bottom four corners were leak tested and only corner R-4 was still leaking. This was a small leak and was not repaired. Environmental conditioning was resumed on November 20.

3.9 Dynamic Load Cycling of Tank No. 3 After 54 Environmental Conditioning Cycles (February 1973)

1. After completion of 54 environmental conditioning cycles (see Table 7) tank no. 3 was removed from the environmental conditioning chamber and installed on the dynamic test device. The tank was load cycled at all three temperatures per Section 3.3.1. The tank was removed and leak tested. Leakage is shown in Appendix B page B-20. There were 26 seam leaks not including one seam on the upper skin which was leaking slightly over almost its entire length of 48 inches. All eight corners were leaking, primarily at the two injections in each corner.

2. The tank was opened and examined. There was no significant change in interior fillet cracks over that previously reported. Interior leakage as indicated by air at 3 psi vacuum bubbling through a water layer is shown in Appendix B, page B-21.

3. The four corners which would be at the bottom of the tank during environmental conditioning (R-1, R-2, R-3, and R-4) were repaired. All sealant in these corners, including the adjacent injections, was removed. The fillets between corners were also removed. Structure was cleaned as previously described and primed with a new sample of Dow Corning 77-123 one-part primer. The primer was air dried 90 minutes at ambient temperature and frozen Dow Corning 77-028 (Lot 40111/, mfg. Aug. 1971) was applied. The sealant was cured at ambient temperatures overnight and then oven cured 1 - 3 hours at 300°F. The corners were leak tested and were leak-free.

4. Laboratory test specimens were removed, tested, and results added to Table 6. Environmental conditioning was resumed on February 19.

3.10 Dynamic Load Cycling of Tank No. 3 After 74 Environmental Conditioning Cycles (August 1973)

1. During the liquid exposure portion of environmental conditioning cycle no. 71 the tank leaked 16 gallons of fuel. The tank was removed from the environmental conditioning chamber and leak tested. One corner at the tank bottom (R-2) appeared to be causing the leakage. This corner was repaired with new sealant using previously described procedures. The door seal was badly cracked and may have been leaking. This seal was replaced with a .125 inch thick fluorosilicone (BMS 1-53) rubber gasket. Environmental conditioning was resumed on June 22.

2. After completion of 74 environmental conditioning cycles (see Table 7) the tank was removed from the environmental conditioning chamber, installed on the dynamic test device and load cycled at all three temperatures per Section 3.3.1. The tank was removed and leak tested. Leakage is shown in Appendix B, page B-22. There were fewer seam and corner leaks than when the tank was tested after 54 environmental conditioning cycles (see 3.9). There were 18 seam leaks and 3 corners leaking. Seam leaks at 5 psi pressure were generally very small. Two of the three corner leaks were at the end of the tank which was up during environmental conditioning. The remaining corner leak was very small. Therefore it was decided not to attempt to repair any of the leaks.

3. The tank was opened and examined. There was no significant change in interior appearance. Interior leakage as indicated by air at 3 psi vacuum bubbling through a water layer is shown in Appendix B page B-23. Only three leaks were visible.

4. Laboratory test specimens were removed from the tank and tested as before. Results were added to Table 6. Environmental conditioning was subsequently resumed the third week in August.

3.11 Dynamic Load Cycling of Tank No. 3 After 92 Environmental Conditioning Cycles (January 1974)

1. After completion of 92 environmental conditioning cycles the tank was removed from the environmental conditioning chamber and leak tested in water. Leakage is shown in Appendix B, page B-24. There was considerably more leakage than when the tank was tested after 74 environmental conditioning cycles. There were approximately 33 seam leaks and 6 corners were leaking. Nine of the 16 injections at the tank corners were leaking. Based on previous experience no attempt was made to repair any leaks.
2. The tank was opened and examined. Most cracks appeared to be gapped open wider than before. Interior leakage is shown on Appendix B, page B-25.
3. In an effort to see if a properly repaired area would again exhibit cracks, the area shown as 1 in Appendix B, page B-25, was stripped and resealed using minimum size fillets. This area is also shown in Figure 18.
4. Laboratory test specimens were removed from the tank and tested as before. Results were added to Table 6.
5. The tank was placed on the dynamic test device and load cycled at all three temperatures per Section 3.3.1.
6. The four corners at the end of the tank which would be down during environmental conditioning (R-1, R-2, R-3, and R-4) were leak tested and were essentially the same as shown in Appendix B, page B-24; therefore, no repairs were made. The tank was returned to the environmental chamber for additional environmental conditioning.

▼ ▼ E

3.12 Dynamic Load Cycling of Tank No. , After 117 Environmental Conditioning Cycles (August 1974)

1. After completion of 117 environmental conditioning cycles the tank was removed from the environmental conditioning chamber and leak tested in water. Leakage is shown in Appendix B, page B-26. There was considerably more leakage than when the tank was tested after 92 environmental conditioning cycles. Two seams were leaking slightly along their entire length. No attempt was made to repair any leaks.

2. The tank was opened and examined. Interior leakage is shown on Appendix B, page B-27.

3. Laboratory test specimens were removed from the tank and tested as before. Results were added to Table 6.

4. The tank was placed on the dynamic test device and load cycled at all three temperatures per Section 3.3.1.

5. The four corners at the end of the tank which would be down during environmental conditioning (R-1, R-2, R-3, and R-4) were leak tested and leakage was not judged severe enough to repair; therefore, no repairs were made. The tank was returned to the environmental chamber for additional environmental conditioning.

3.13 Dynamic Load Cycling of Tank No. 3 After 129 Environmental Conditioning Cycles (December 1974)

1. During the liquid portion of the third environmental conditioning cycle (no. 120), 11.5 gallons of fuel leaked into the environmental chamber. The tank was removed and examined. Two lower corners (F-1 and F-2) were leaking extensively. These corners were repaired with new sealant. The tank was then returned to environmental conditioning.

2. After completion of 129 environmental conditioning cycles the tank was removed from the environmental conditioning chamber and load cycled on the dynamic test device at all three temperatures per 3.3.1. The tank was then leak tested in water. Leakage is shown in Appendix B, page B-28. There was considerably more leakage than when the tank was tested after 117 environmental conditioning cycles. Six of the eight longitudinal seams were leaking over much of their entire length. Fifteen of the 16 injections at the tank corners were leaking. All eight corners were leaking. The tank was opened and examined. Interior leakage is shown on Appendix B, page B-29.

3. Laboratory test specimens were removed from the tank and tested as before. Results were added to Table 6.

4. Testing of this tank was considered completed at this point.

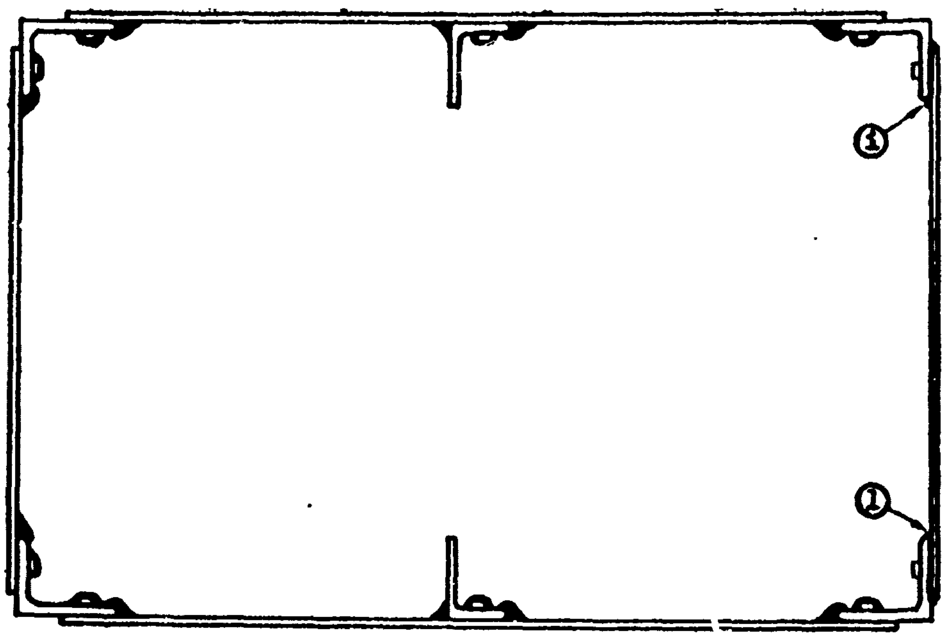
4. REFERENCES

1. ML-TDR-64-207, High Temperature Elastomeric Integral Fuel Tank Sealant Evaluations.

2. Boeing Drawing 35-22615, Tank Assembly-Sealant Test.

3. BAC 5504, Integral Fuel Tank Structure Sealing.

4. Boeing Drawing 65A14621, Tank Assembly Sealant Test. A



① Faying Surface Seals.

FIGURE 1 - TYPICAL CROSS-SECTIONAL VIEW OF FILLET SEALED TANK NO. 1

FIGURE 2 - PARTIAL ASSEMBLY AND SEALING OF TANK NO. 1

BW-173623

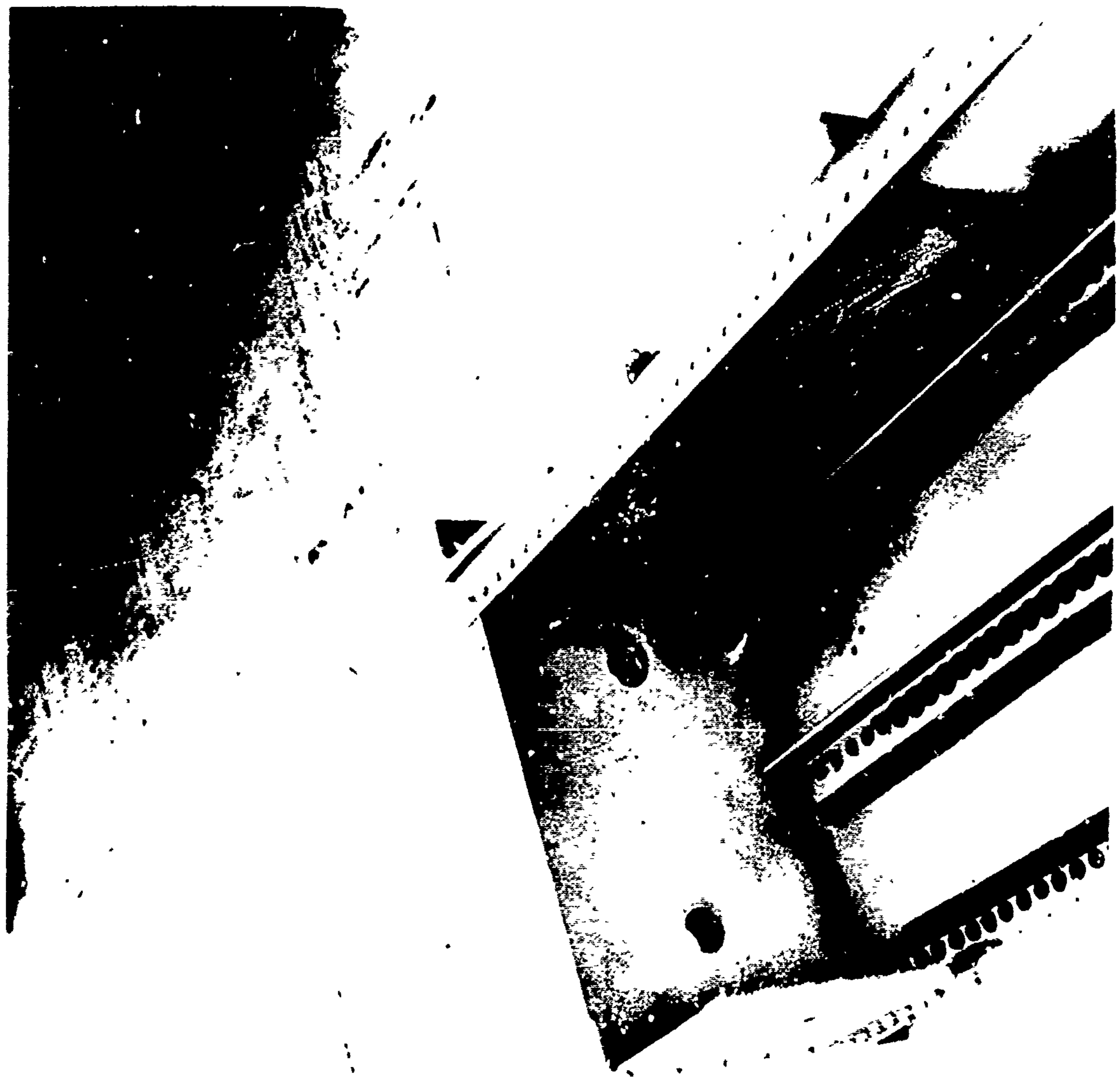


FIGURE 3 - PARTIAL ASSEMBLY AND SEALING OF TANK NO. 1 - UPPER END VIEW

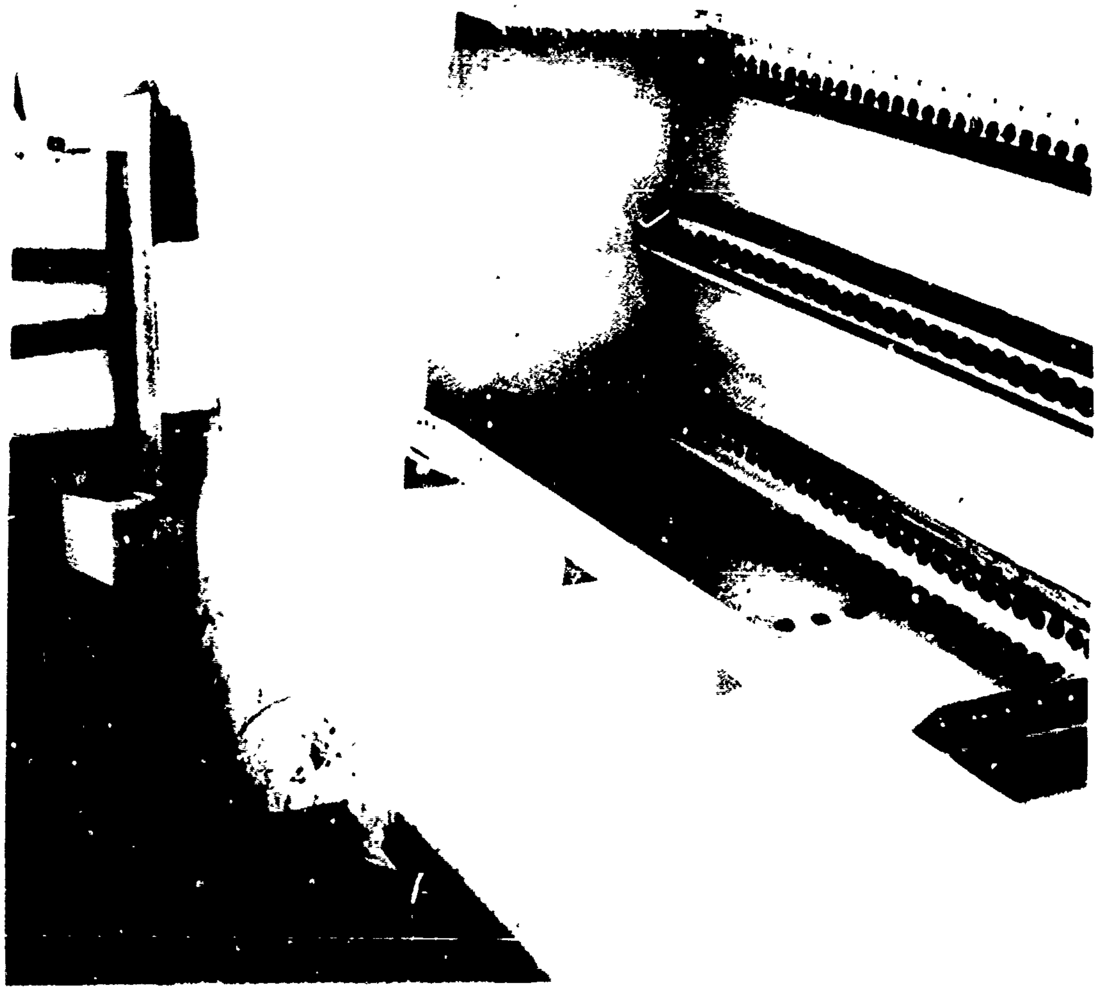
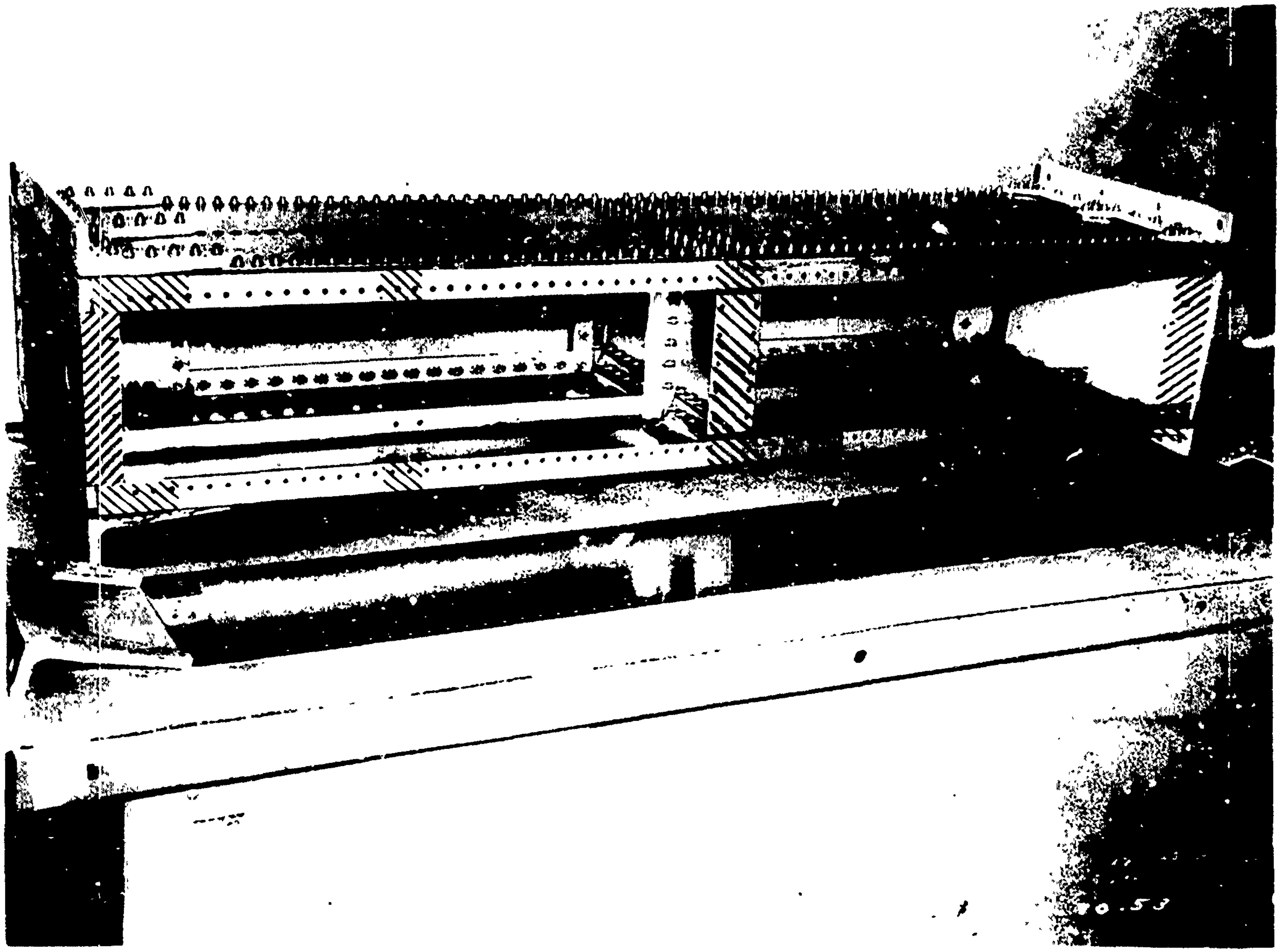


FIGURE 4 - PARTIAL ASSEMBLY AND SEALING OF TANK NO. 1 - LOWER END VIEW

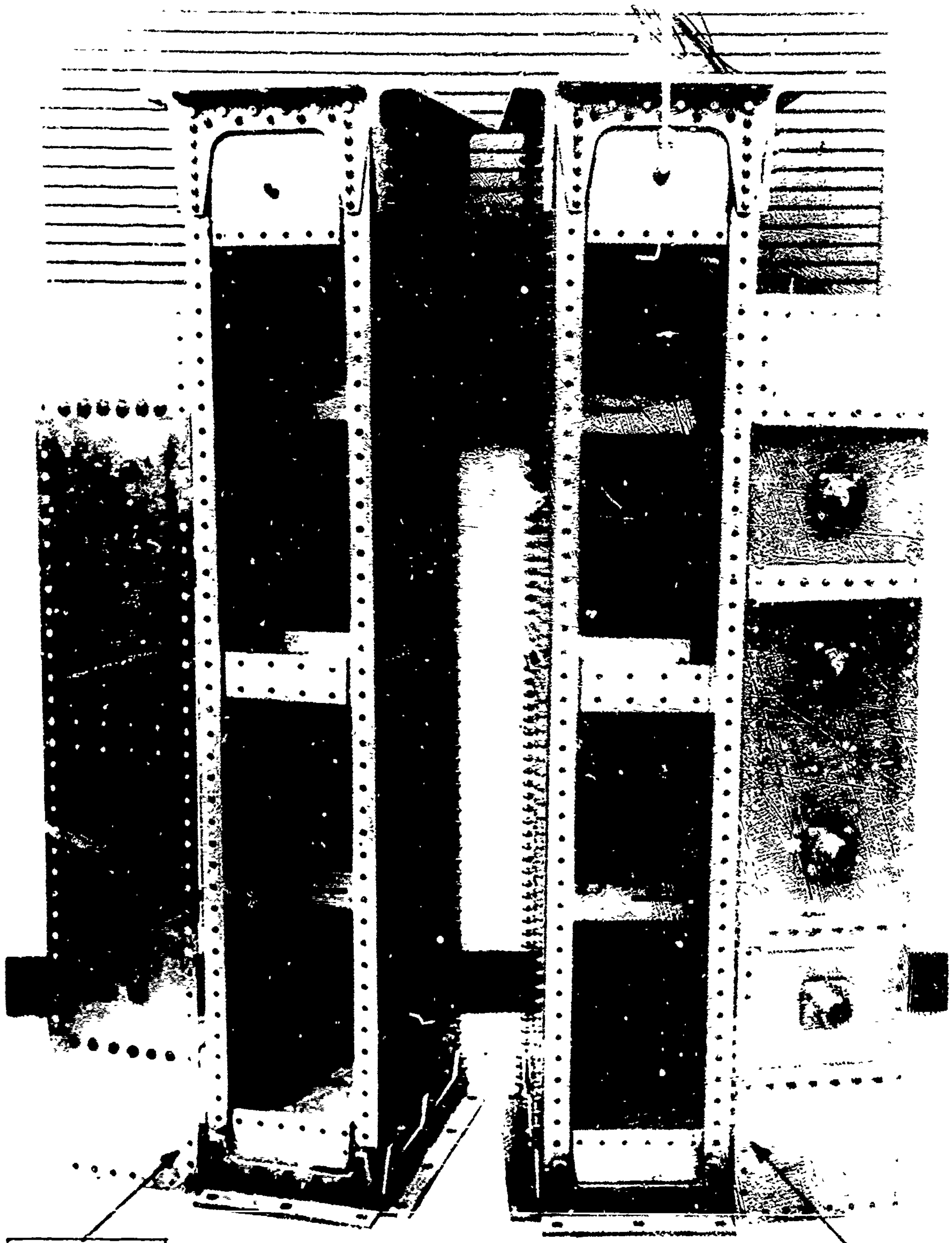
BW-173625



NOTE: "Block-Off" faying surface seals were added to upper and lower skin and the spar web of Tank No. 1, as shown typically by \\\\\\, prior to the installation of the lower skin.

FIGURE 5 - PARTIAL ASSEMBLY AND SEALING OF TANK NO. 2

BW-173626



Tank No.1 FIGURE 6 - COMPLETED TEST TANKS Tank No.2

Arrows indicate thermocouple locations. BW-173627

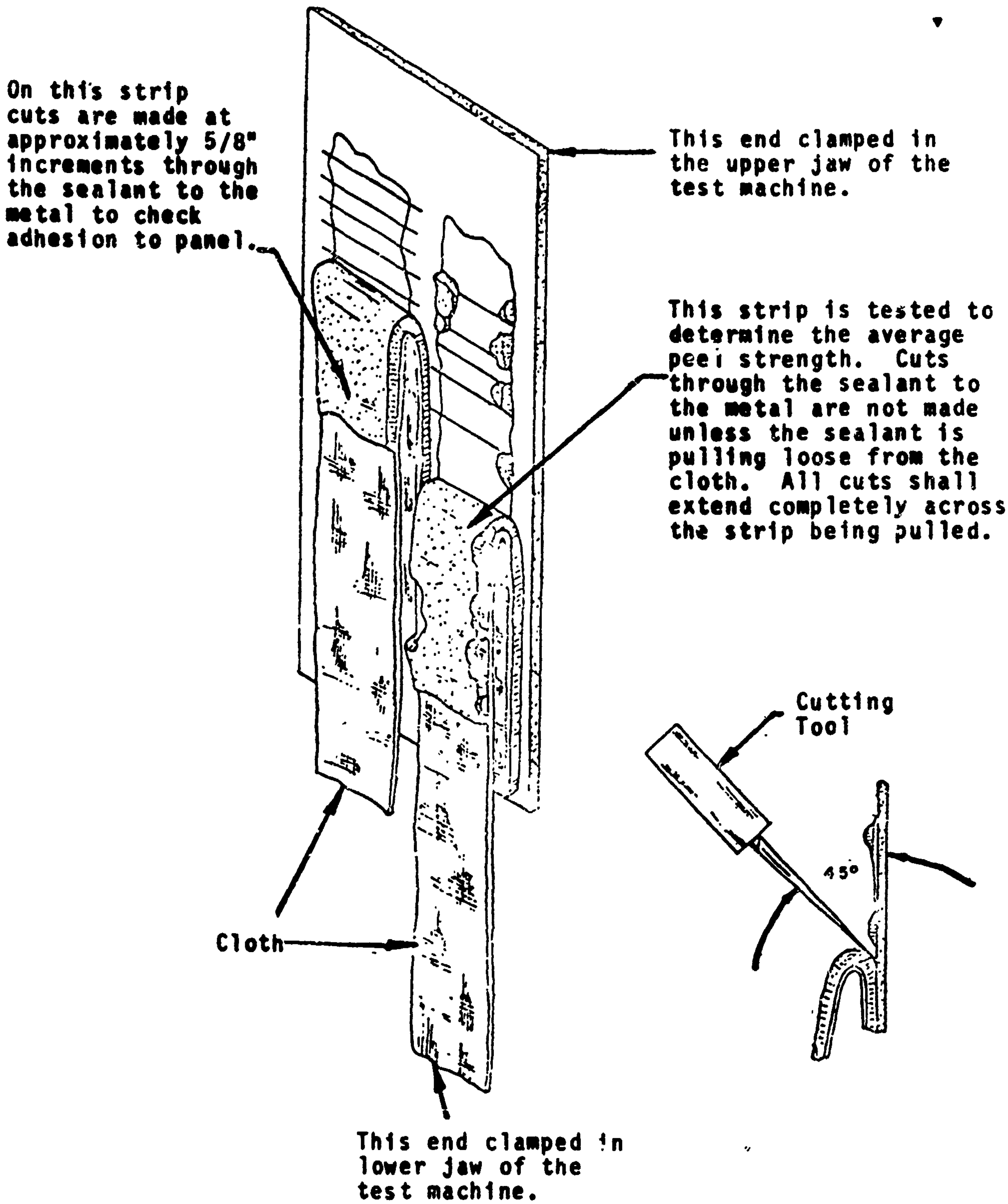
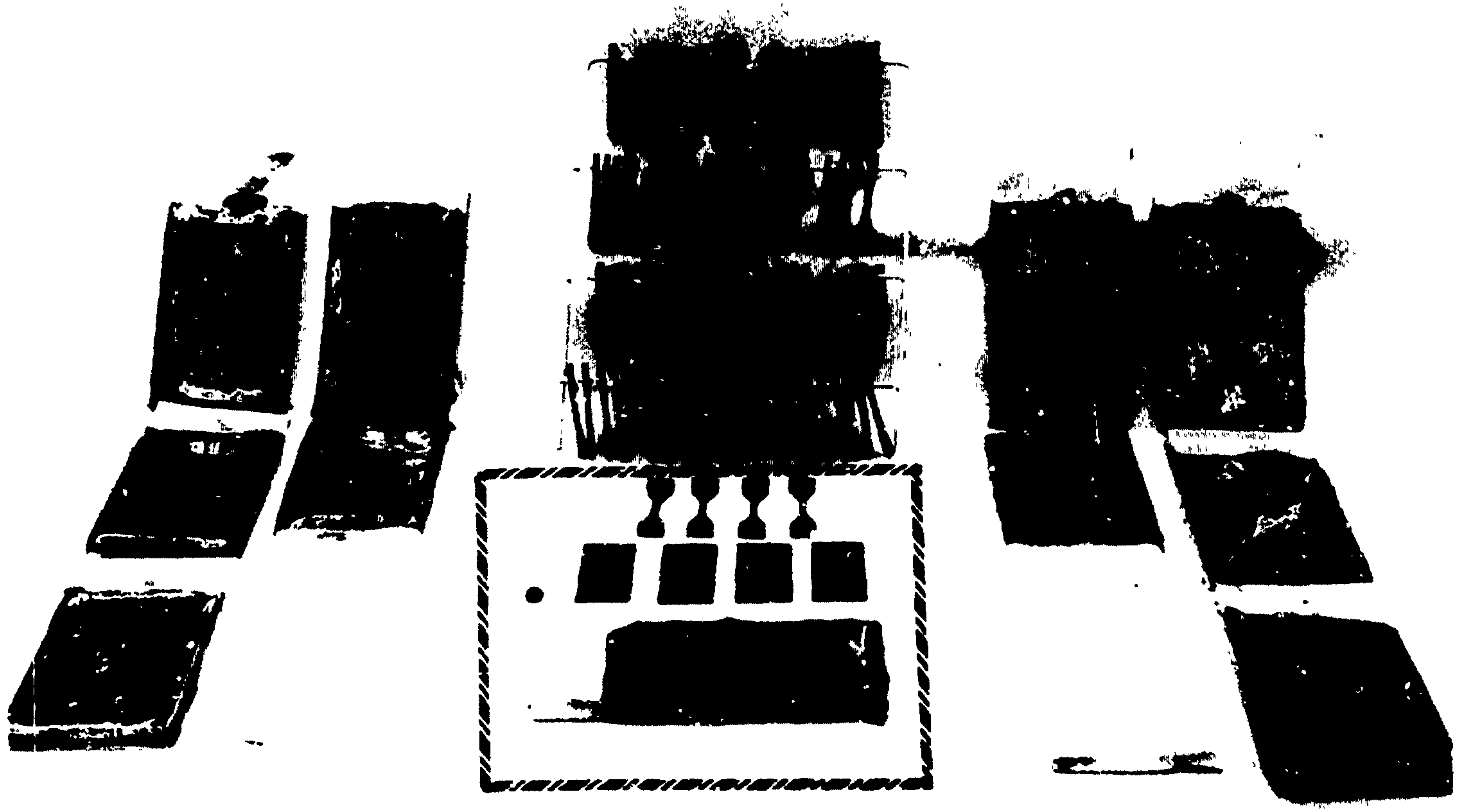


FIGURE 7 - PEEL STRENGTH TESTING



• A typical set of test specimens.

FIGURE 8 - TEST SPECIMENS READY FOR INSTALLATION IN ONE TEST TANK

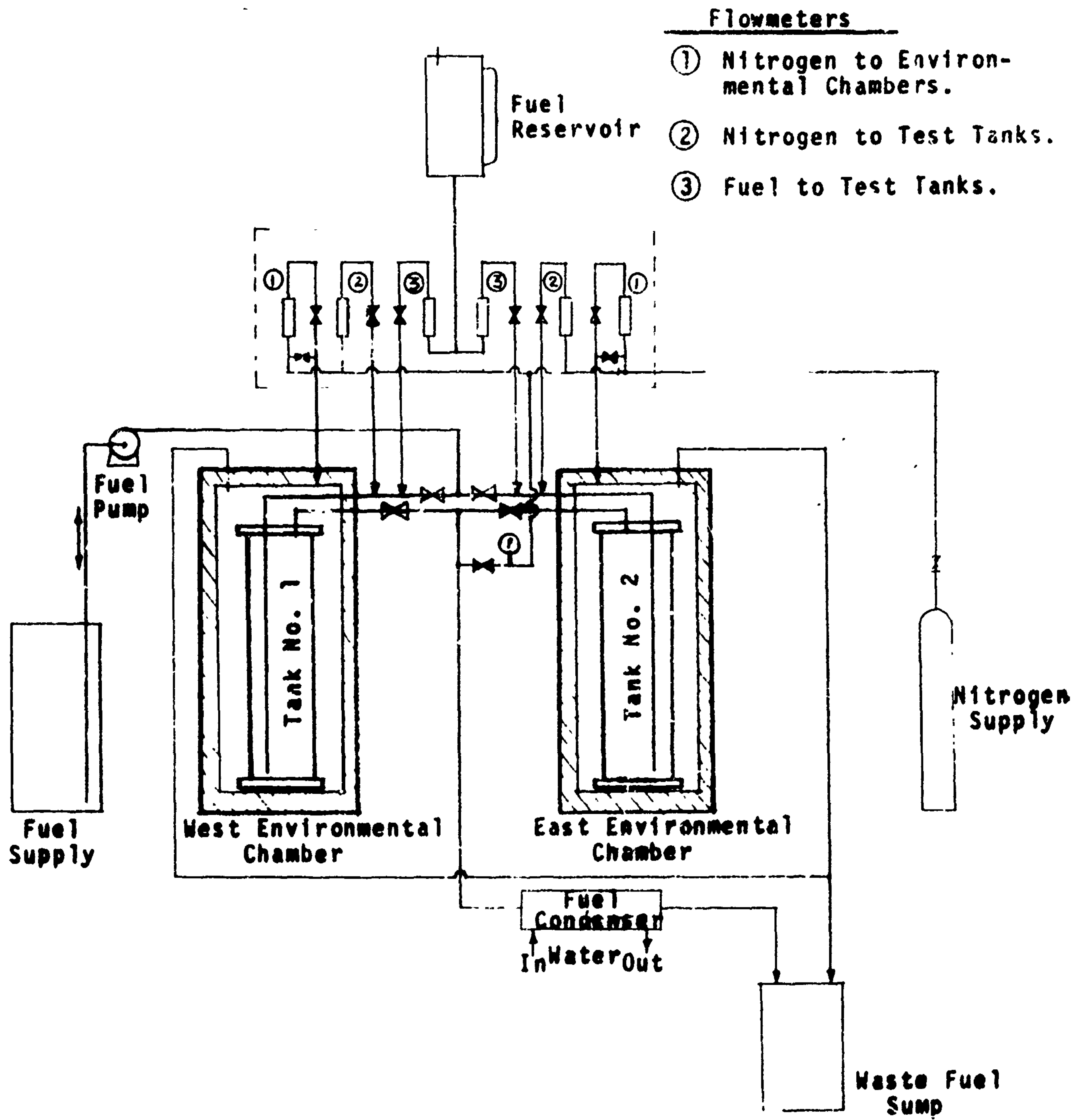


FIGURE 9 - FLOW CHART - ENVIRONMENTAL CONDITIONING OF TEST TANKS

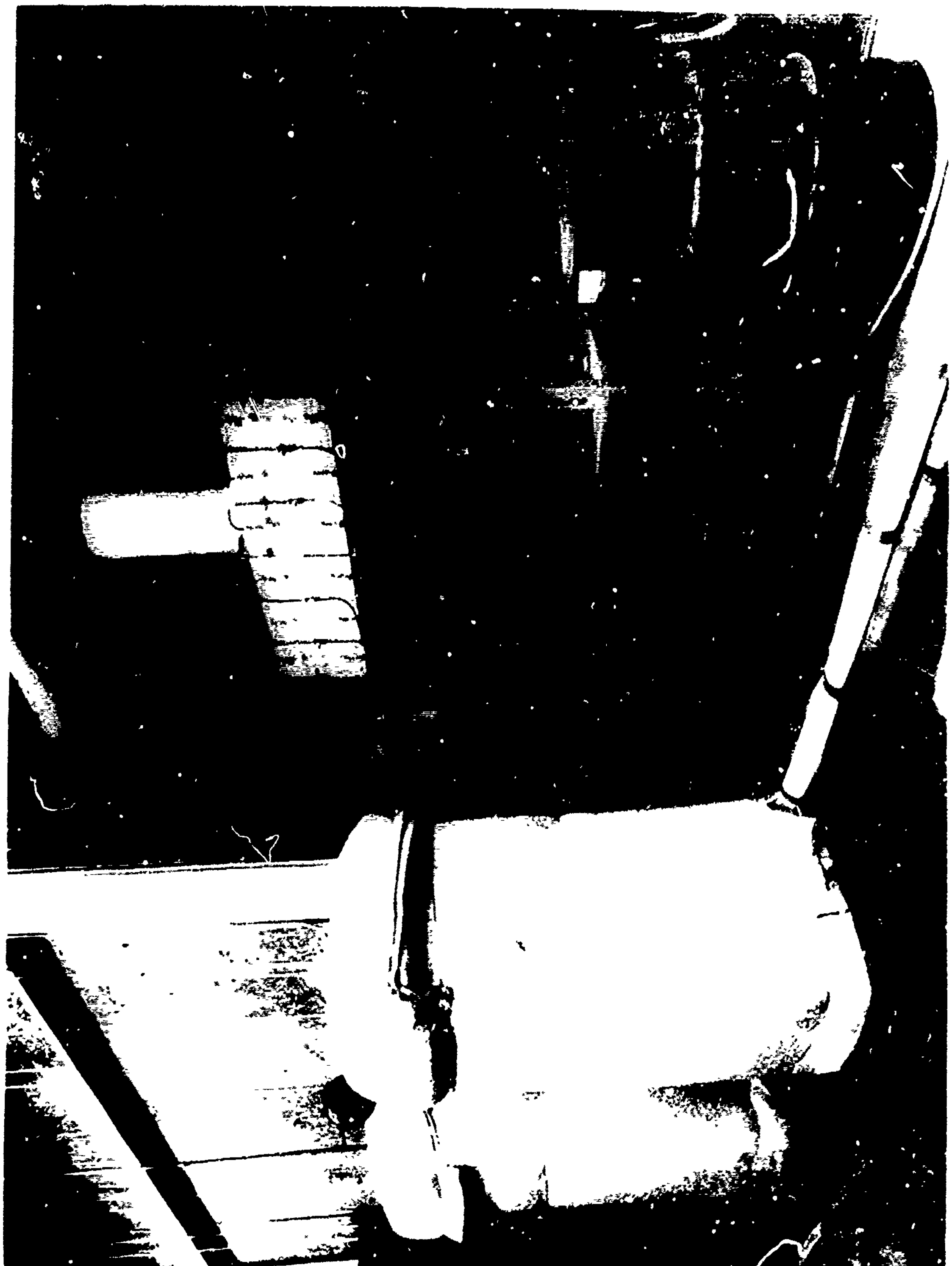


FIGURE 10 - ENVIRONMENTAL CONDITIONING TEST EQUIPMENT

FIGURE 11 - ENVIRONMENTAL CHAMBER WITH SIDE HEATERS INSTALLED

BW-173629

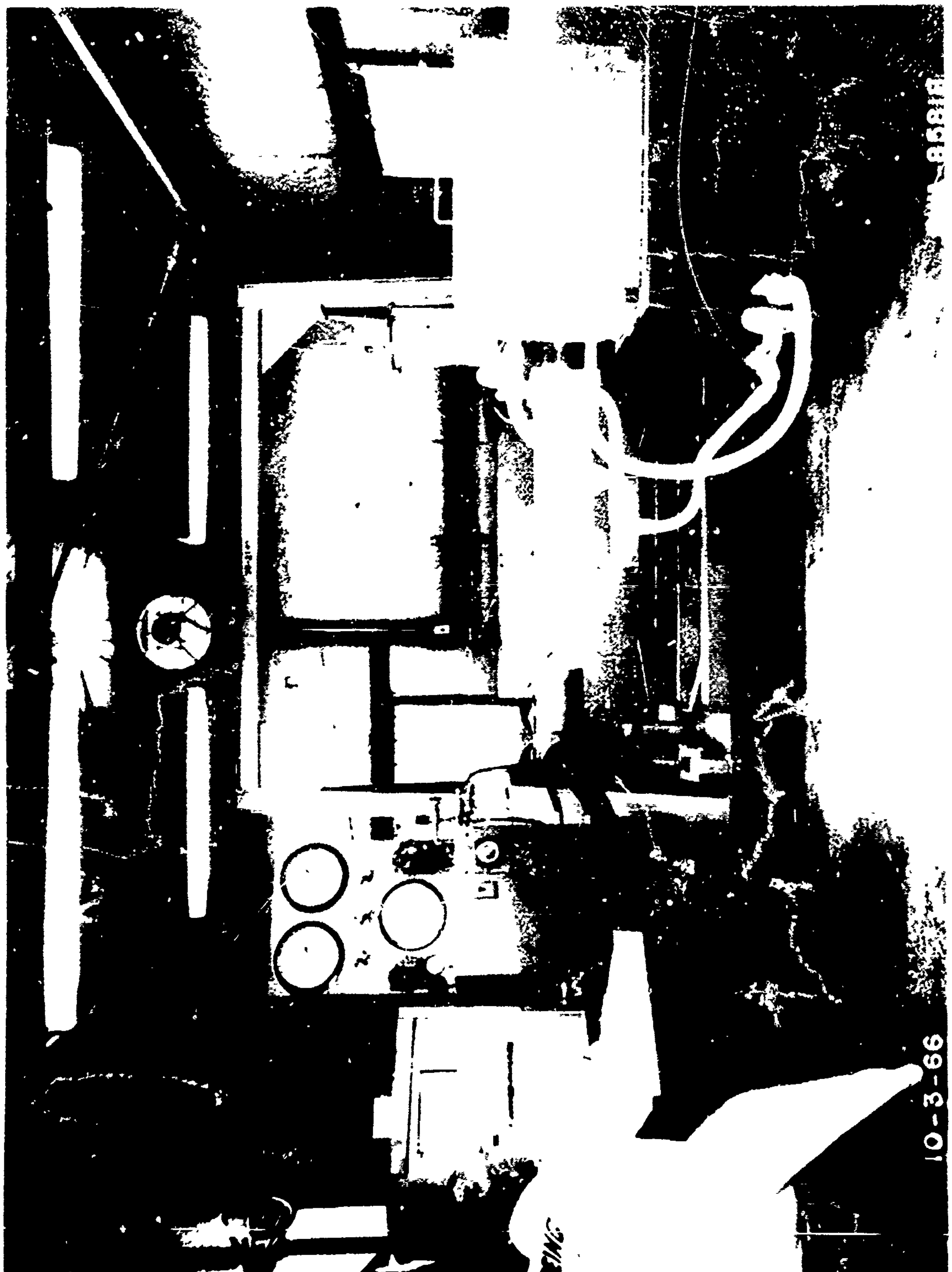


FIGURE 12 - DYNAMIC TEST DEVICE (WITH CIRCULATING COLD FUEL)

A

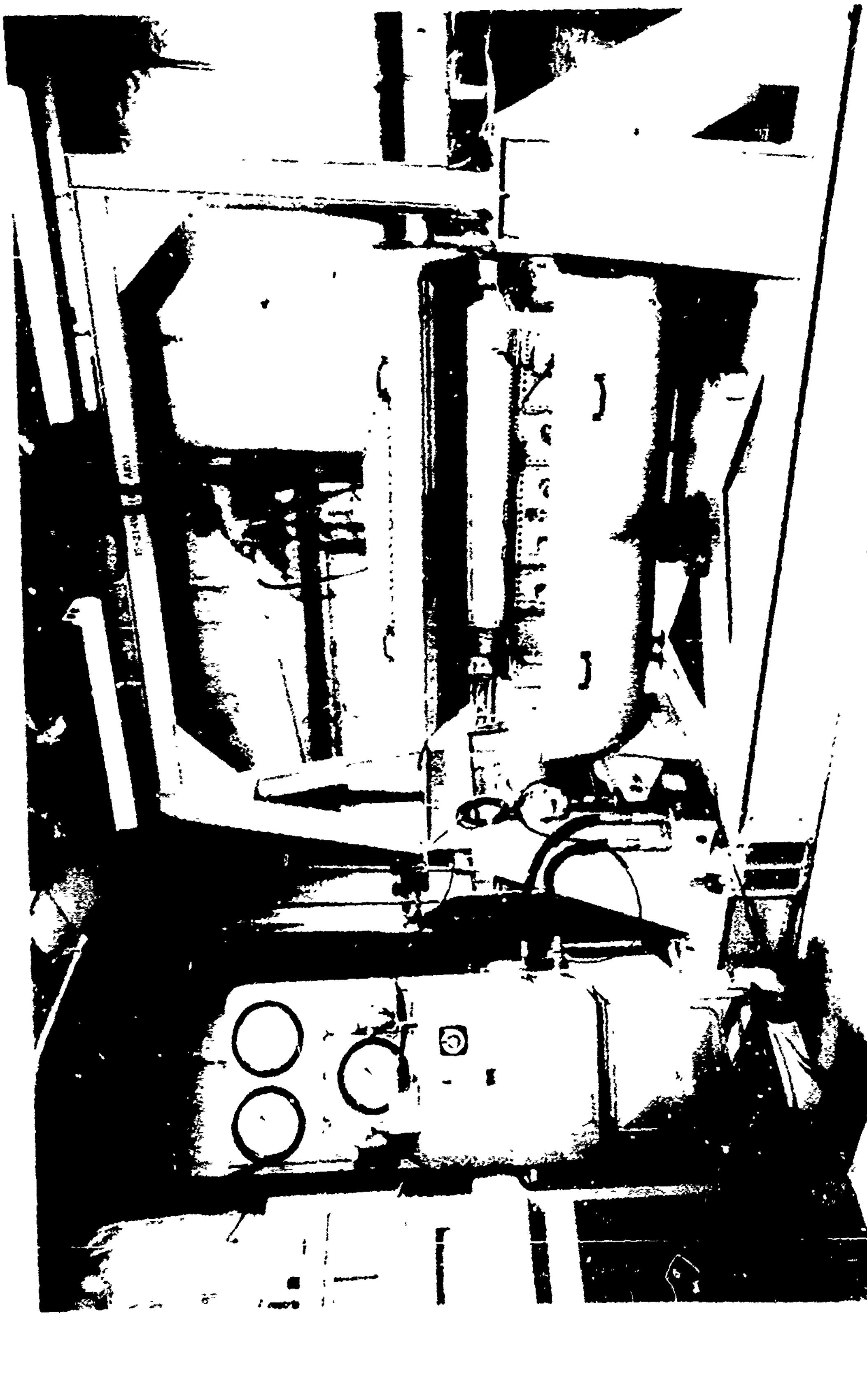


FIGURE 13 - DYNAMIC TEST DEVICE (WITH INSULATING CHAMBER FOR HOT AIR CIRCULATION)

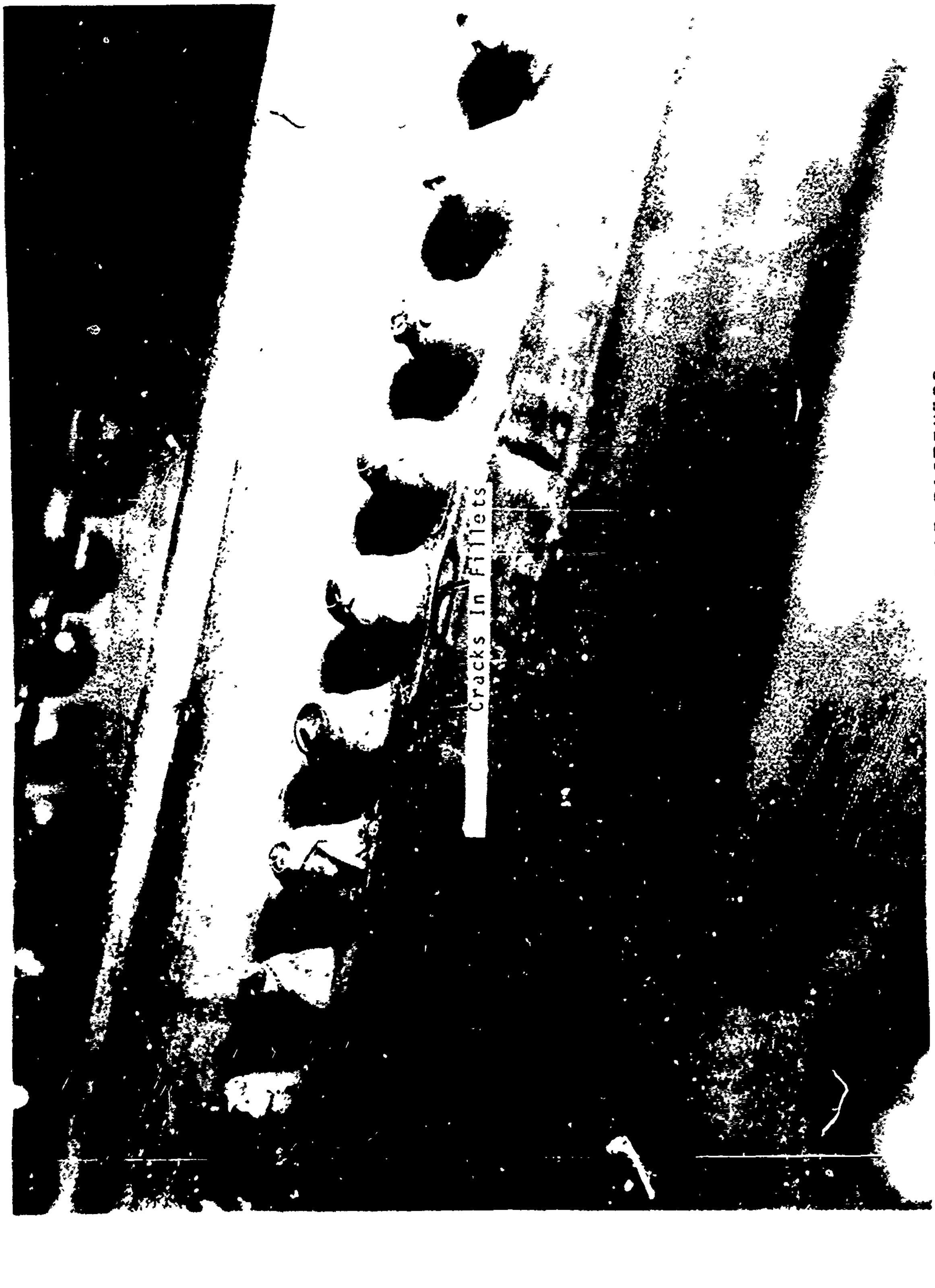


FIGURE 14 - CRACKS IN FILLETS AT BASE OF FASTENERS

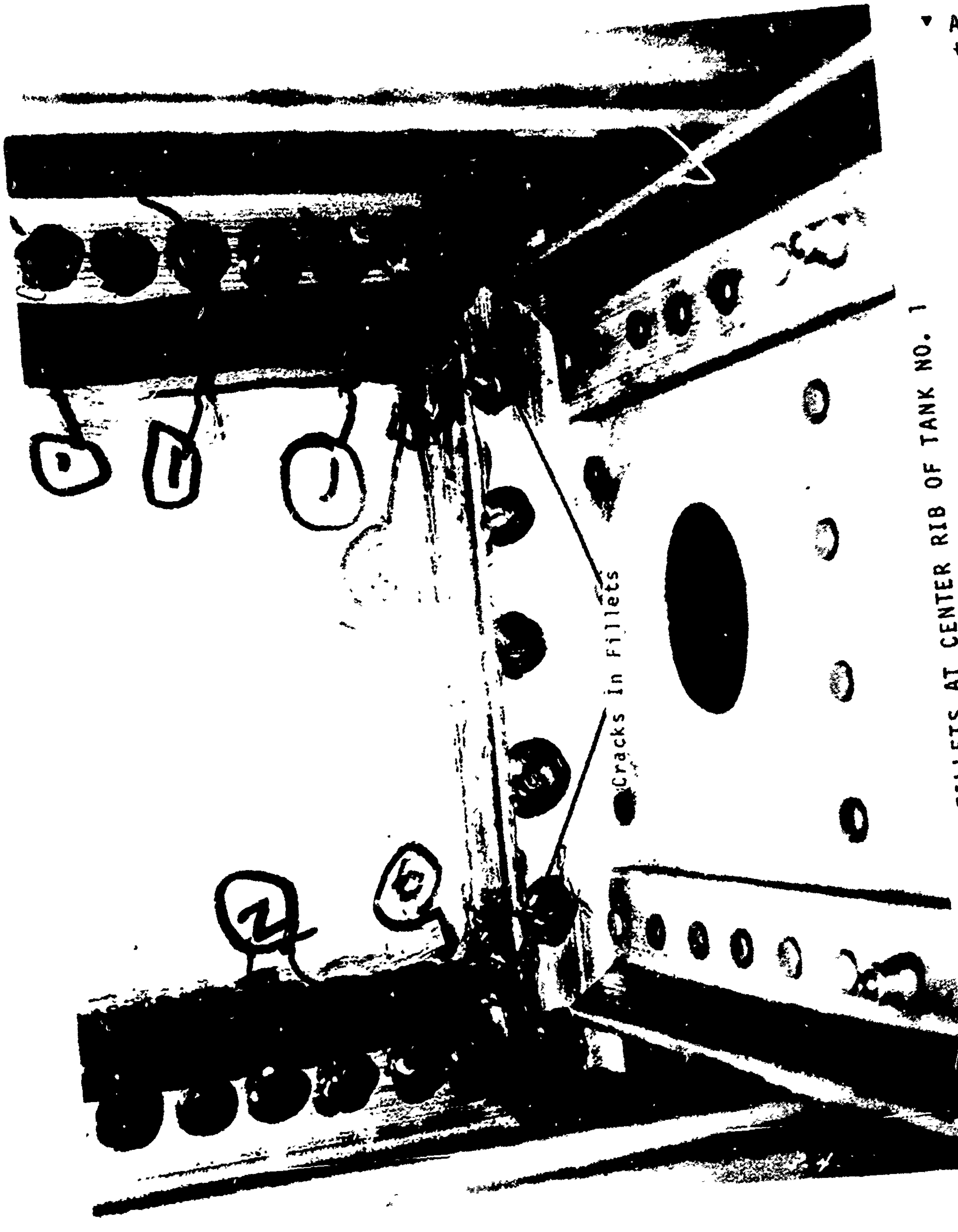


FIGURE 15 - CRACKED FILLETS AT CENTER RIB OF TANK NO. 1

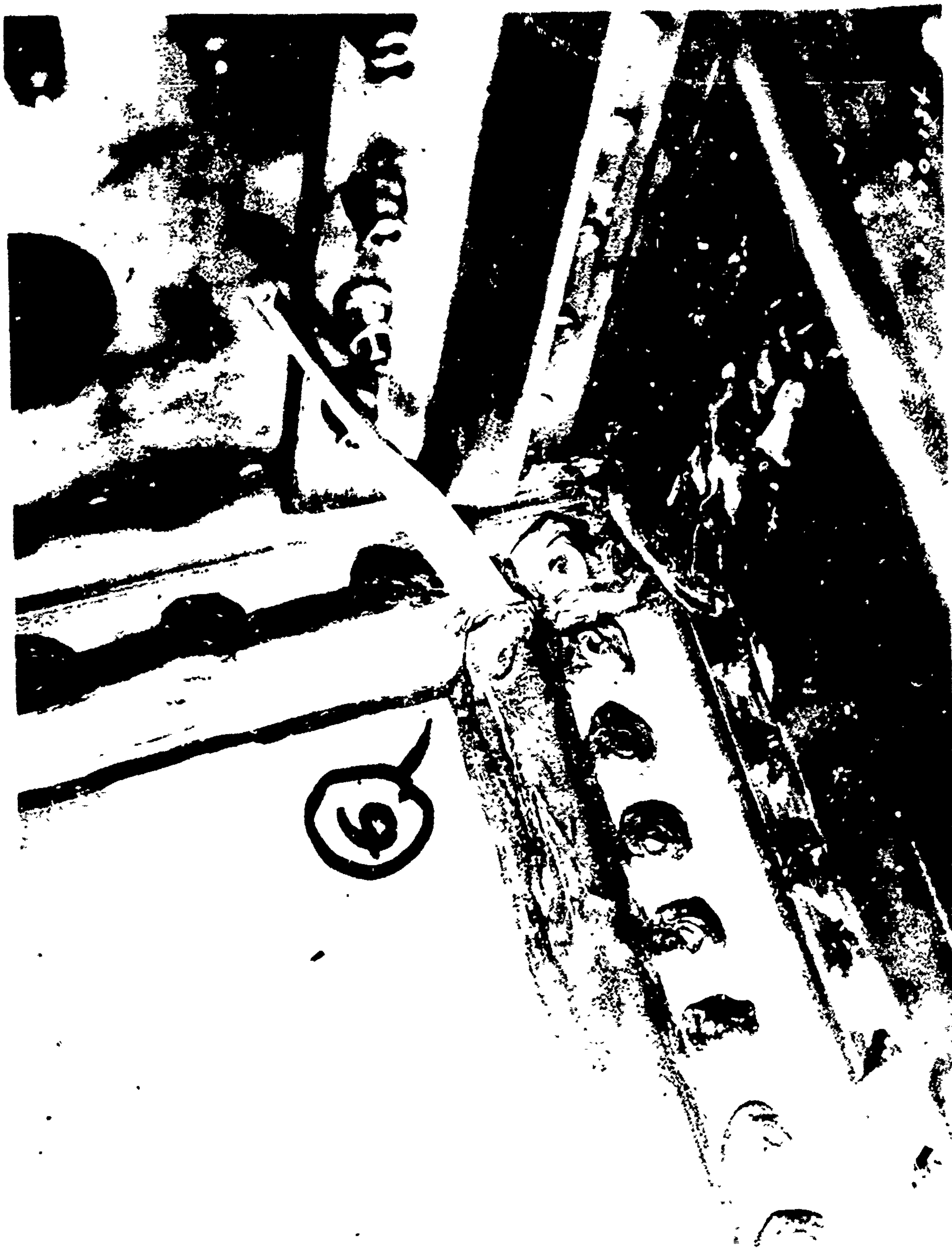


FIGURE 16 - CRACKED FILLET AT CENTER RIB OF TANK NO. 2

FIGURE 17 - FILLET AT CENTER RIB OF TANK NO. 2
SHOWING CRACK AND LOSS OF ADHESION

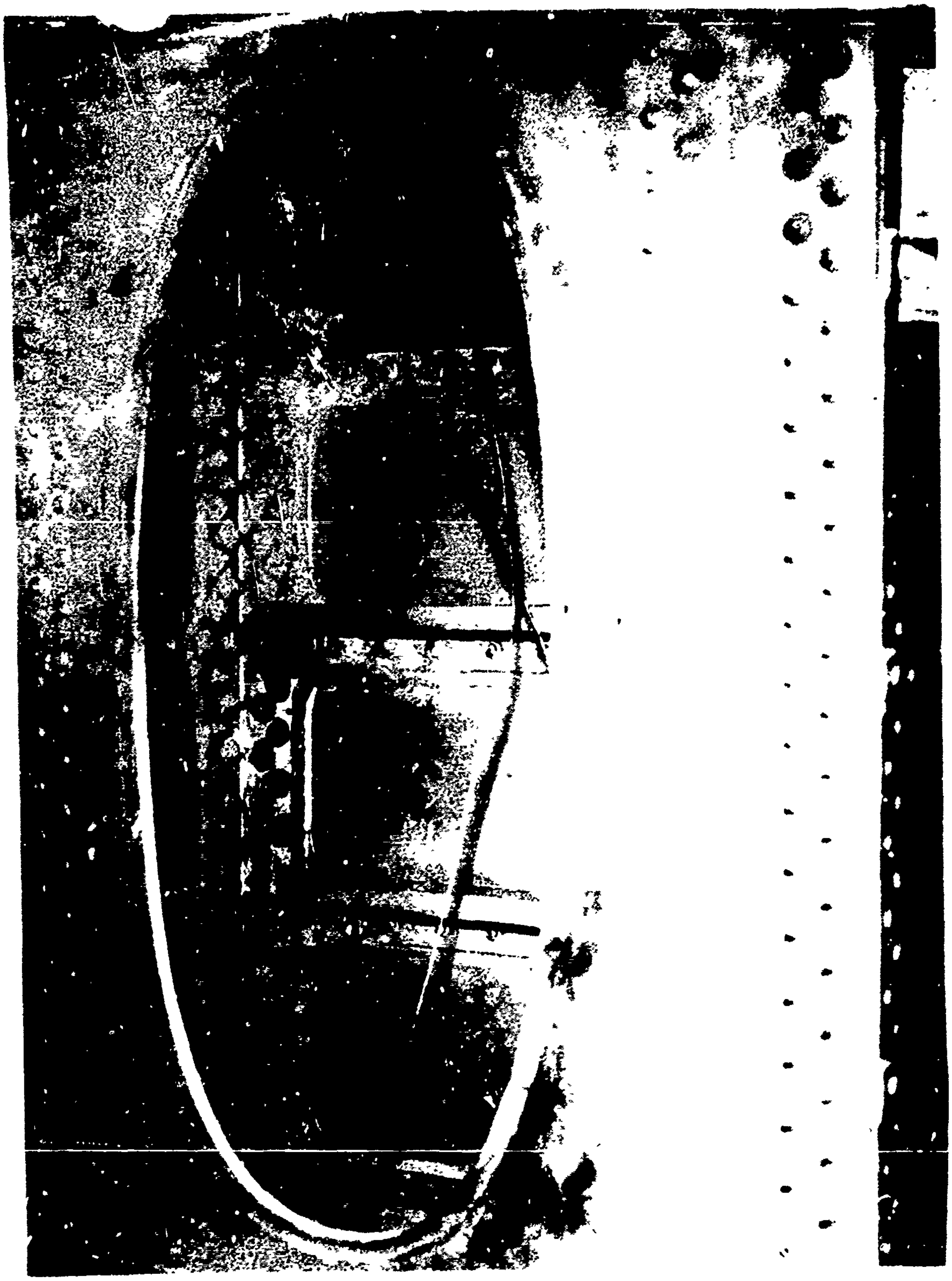


FIGURE 18 - AREA OF TANK NO. 3 REPAIRED WITH SMALL FILLETS IN FEBRUARY 1974

TABLE 1
PHYSICAL PROPERTY TEST RESULTS FOR TANK NUMBERS 1 AND 2

Physical Property (4 Specimens per Condition)	Test Temp., °F	Control Specimens (No Env. Exposure)	Environmental Conditioning Cycles Completed (1)				
			4 Cycles		12 Cycles	20 Cycles	
			Tank No. 2	Tank (4) No. 1	Tank (5) No. 1	Tank (5) No. 1	Tank No. 1
Tensile Strength, psi Avg. (Range)	75 ±5	603 (351)	454 (118)	414 (145)	354 (130)	310 (77)	476 (139)
psi Avg. (Range)	440 ±10	277 (101)	170 (16)	236 (105)	--- ---	--- ---	--- ---
Ultimate Elongation, % Avg. (Range)	75 ±5	196 (125)	95 (20)	113 (55)	86 (15)	91 (25)	117 (25)
psi Avg. (Range)	440 ±10	100 (45)	69 (15)	78 (10)	--- ---	--- ---	--- ---
Peel Strength, Lb./In. Avg.	75 ±5	13	2.5	2.5	2	2.5	2.5
(3) % Cohesive Separation		100	95	60	50	98	90
Volume Change, % Avg.	75 ±5	---	+4.5	+1.9	+0.2	-1.3	+0.6
Weight Change, % Avg.	75 ±5						
Fuel Soaked		---	+2.9	+1.1	-1.2	-2.1	-0.3
Dried Out (2)		---	-1.9	-1.7	-4.0	-5.1	-3.1
Durometer Hardness, pts Avg.	75 ±5						
Initial (Range)		---	48 (2)	42 (9)	46 (5)	42 (3)	46 (4)
Fuel-Soaked (Range)		---	49 (2)	49 (2)	49 (1)	51 (4)	50 (2)
Dried-Out (2) (Range)		---	50 (2)	50 (2)	51 (2)	54 (3)	53 (2)

(1) Hours in fuel vapor at 4415-441°F: 4 Cycles 524 Hours; 12 Cycles 1564 Hours; 20 Cycles 2620 Hours

(2) Circulating Air Oven 440 ±10°F for 24 Hours.

(3) One Panel per Condition.

(4) All specimens immersed in liquid fuel at room temperature for one day prior to testing.

(5) These specimens were in Tank No. 2 during the first 4 cycles of Environmental Conditioning

C

TABLE 2

ENVIRONMENTAL CONDITIONING CYCLES FOR TANK NO. 1

Environmental Conditioning Cycle No.	Liquid Fuel Exposure Hours				Fuel Vapor Exposure Hours					
	Ambient to 139°F	140 to 150°F	Over Temp. 151°F to +°F	Over Temp. Hrs.	Ambient to 400°F	401 to 425°F	415 to 441°F	426 to 441°F	Over Temp. (1) 442°F to +°F	Over Temp. (1) Hrs.
1	169.0				16.0	18.0	133.0		457	.4
2	6.0	16.0			2.0		138.5			
3	1.6	20.0					138.5			
4	5.0	10.5	152	6.0			114.0			
5	16.5	2.0			18.5	5.0		165.5	443 445	4.0 1.0
6	1.0	21.0	152	2.0	8.0	3.0	10.0	124.8	444	6.5
7		15.5	175	5.0	3.0	1.0	5.0	113.0		
8		17.0	160	4.0	5.5	1.5	11.0	128.5		
9 (2)		15.5	160	5.5	28.5	1.3		90.0	443	.3
10	2.0	13.5	165	8.0	4.5	1.0	19.0	146.5	443	5.0
11	1.5	20.0			6.5	1.5	21.0	90.8	450	1.5
12	2.0	22.0			25.5	19.0	34.5	80.0	445	6.5
13	5.0	16.5			3.5	1.8	9.5	105.8		
14	16.5	5.0	160	2.0	52.5	3.0		89.5		
15	1.0	17.0	157	3.0	23.3	3.8		115.3	446	1.0
16	2.0	17.5	175	2.0	3.3	1.3	2.0	140.0	448	.5
17	1.0	18.5	175	3.0	3.0	.8	1.0	140.8	443	1.0

(1) Hours in this column are included in hours reported at 415-441°F or 426-441°F.

(2) Started using fuel from Standard Oil of California.

(Continued on next page)

A

TABLE 2 (Continued)

ENVIRONMENTAL CONDITIONING CYCLES FOR TANK NO. 1

Environmental Conditioning Cycle No.	Liquid Fuel Exposure Hours				Fuel Vapor Exposure Hours						
	Ambient to 139°F	140 to 150°F	Over Temp. 151°F to ↓°F	Over Temp. Hrs.	Ambient to 400°F		401 to 425°F	415 to 441°F	426 to 441°F	Over Temp. (1) 442°F to ↓°F	Over Temp. (1) Hrs.
					Under 140 °F (5)	140°F to 400°F (5)					
18	1.0	7.5	175	13.0	5.0		3.3		138.8	446	0.5
19	3.0	17.5			4.5		1.5		140.5		
20	2.5	18.0	175	0.5	2.5		0.3		173.3	4.5	14.0
21	336.0 (4)				2.5		1.3		140.0	443	8.0
22 (3)	1.0				30.0		1.8		304.5	445	0.3
23	1.0				29.0	18.5	2.0		187.0		
24	1.5				52.5	30.5	4.5		100.5		
25	1.5				[illegible]	20.5	2.5		93.0		
26	1.0				19.5	32.0	6.5		94.5		
27	1.0				42.5	45.5	9.0		237.5		
28	1.0				8.0	17.0	6.5		158.5		
29	1.5				35.0	37.0	6.0		92.0		
30	1.0				69.0	65.0	5.0		370.5		
31	1.0				4.0	37.0	5.5		101.0		
TOTALS	584.1	290.5	151-175°F	53.0	824.1		117.7	63.7	3862.[illegible]	442-457°F	50.5 (1)

(1) Hours in this column are included in hours reported at 415-441°F or 426-441°F.

(3) Started using short liquid fuel cycle from here on due to excessive leakage.

(4) This was a 14 day exposure at ambient temperature

(5) Most of the hours in these columns are due to unplanned cool downs caused by over temperature controls turning heaters off.

A

TABLE 3

ENVIRONMENTAL CONDITIONING OF TANK NO. 2

Environmental Conditioning Cycle No.	Liquid Fuel Exposure Hours				Fuel Vapor Exposure Hours		
	Ambient to 139°F	140 to 150°F	Over Temp. 150°F to ↓°F	Over Temp. Hrs.	Ambient to 400°F	401 to 425°F	415 to 441°F
1	169.0				16.0	18.0	133.0
2	6.0	16.0			2.0		138.5
3	1.6	20.0					138.5
4	2.0	9.5	155	10.0			114.0
Total	178.6	45.5	150-155°F	10.0	18.0	18.0	524.0
	234.1 Hours				560 Hours		

TABLE 4

LEAKAGE OF TANK NUMBERS 1 AND 2
AFTER FOUR CYCLES OF ENVIRONMENTAL CONDITIONING

Tank No.	Location Of Leakage*	Number Of Leaks			
		After 4 Cycles Of Environmental Conditioning		After 4 Cycles Of Environmental Conditioning Plus 510 Hot Load Cycles Per Section 2.4.3	
		Joint Leaks	Fastener Leaks	Joint Leaks	Fastener Leaks
1	Primary Test Areas				
	Upper Skin	0	8	0	11 ①
	Lower Skin	0	2	0	7
	Spar	0	1	0	4
	Secondary Test Areas				
	Access Door	8	0	Many	Many
	Ends Of Tank	≈13	2	11	29
2	Primary Test Areas				
	Upper Skin	0	2	14	20 ②
	Lower Skin	0	3	0	5
	Spar	0	1	1	≈26
	Secondary Test Areas				
	Access Door	6	12	Many	Many
	Ends Of Tank	10	3	11	28

① ≈8 leaks in one area. ② ≈5 leaks in one area.

*See Appendix A for specific locations of leaks.

TABLE 5

SUMMARY OF LEAKAGE OF TANK NO. 1 IN PRIMARY TEST AREAS

Type Of Leak	Numbers Of Leaks After Various Cumulative Exposures (See Appendix A For Leak Locations) Leakage Test No.* 1	2	3	4	5	6	7
Fastener Leaks	11	22	26	12	80	89	63
Joint Leaks (Total)	0	0	0	4	10	(3)	(4)
Joint Areas Leaking (1)	0	0	0	4	7	8	6

*Leakage Test No.	Environmental Conditioning Cycles Completed	Total Fuel Vapor Exposure At 415 - 441°F	Total Dynamic Load Cycles Completed, Test Temperature 440°F	-50°F	Ambient
1	4	524 Hours	0	0	0
2	4	524 Hours	510	0	0
3 (2)	4	524 Hours	510	0	0
4	4	524 Hours	510	510	510
5	4	524 Hours	560	510	510
6	12	1564 Hours	560	510	510
7	12	1564 Hours	1070	1020	1020

(1) There are 24 joint areas, separated by faying surface seals, in the primary test area (8 each on top skin, bottom skin, and spar).

(2) Same exposures as Leakage Test No. 2, except after repair of loose and/or cracked fillets.

(3) Eight leaks in four joint areas, plus continuous joint leakage along entire lengths of four additional joint areas.

(4) Eight leaks in five joint areas, plus continuous joint leakage along entire lengths of two additional joint areas.

TABLE 6
PHYSICAL PROPERTY TEST RESULTS FOR TANK NUMBER 3

Physical Property	Test Temp. °F (1)	Control Specimens (No Environmental Exposure)	Environmental Conditioning Cycles Completed (2)							
			8 Cycles	22 Cycles	39 Cycles	54 Cycles	74 Cycles	92 Cycles	117 Cycles (5)	129 Cycles
Tensile Strength, psi Avg. (Range)	75 450 -50	675(90) (4) 227(104) (5) 1701(159) (5)	667(198) 197(90) 2058(308)	556(33) 94(44) ------	429(243) 113(51) -------	254(107) 101(20) --------	233(57) 96(29) -------	227 (59) 61 (24) (7) 80 (47)	160 (48) 39 (24) --	200 (47) 91(114) 607(132)
Ultimate Elongation, % Avg. (Range)	75 450 -50	265(20) (4) 30(20) (5) 350(45) (5)	150(40) 75(20) 23[illegible](40)	145(10) 77(10) --------	147(45) 63(25) -------	117(15) 62(20) -------	74(10) 46(10) -----	78 (20) 34 (25) (7) 39 (20)	43 (10) 17 (5) --	44 (10) 18 (5) 54 (15)
Peel Strength, lb./in. Avg. % Cohesive Separation	75 75	---- ----	7 10%	6.5 80% (6)	5 80%	6 100% (6)	-----	-----	--- ---	12 (8) 85 (6)
Volume Change, % Avg.	75	----	-0.7%	-1.7%	-3.9	-3.4%	-8.8%	-10.1%	-14.3%	-13.8% (5)
Weight Change, % Avg. Fuel Soaked	75	----	-1.5% ----	-2.2%	-2.8%	-4.0	-8.2%	-9.1%	-10.6%	-12.3% (5)
Durometer, Hardness PTS Avg.	75	39 (2) (3)	45	46	42	43	49	51	55	58

(1) Temperature Tolerance is ±5°F.
(2) Per Table 7.
(3) Average and Range For All 26 Specimens.
(4) Only 2 Specimens Per Condition.
(5) Only 3 Specimens Per Condition.
(6) Not true cohesive separation. Separation was adjacent to wire screen tab.
(7) At 438°F.
(8) Before peel testing, this panel has been exposed to ambient laboratory conditions for about 3 months (after environmental conditioning).

TABLE 7
ENVIRONMENTAL CONDITIONING OF TANK NUMBER 3

Environmental Conditioning Cycle No.	Fuel Liquid Exposure Hours: Ambient To 139°F	Fuel Liquid Exposure Hours: 140 To 150°F	Fuel Liquid Exposure Hours: Over Temperatures: 151°F To +°F	Fuel Liquid Exposure Hours: Over Temperatures: Hours	Fuel Vapor Exposure Hours: Below 140°F (1)	Fuel Vapor Exposure Hours: 140 To 400°F (1)	Fuel Vapor Exposure Hours: 401 To 425°F	Fuel Vapor Exposure Hours: 415 To 441°F	Fuel Vapor Exposure Hours: 426 To 441°F	Fuel Vapor Exposure Hours: Over Temperatures: 441°F To +°F	Fuel Vapor Exposure Hours: Over Temperatures: Hours (3)
1	4.0	15.0				19.5	5.5		100.0		
2	18.5					3.3	2.5		115.8		
3	4.0	16.5			16.0	32.0	7.5		92.5		
4	5.0	12.5			36.0	35.0	14.5		227.0		
5	2.5	12.0	155	8.0	2.0	4.5	7.[illegible]		156.0		
6	3.0	12.5	156	6.0	58.0	3[illegible].0	6.0		75.0		
7 (2)	5.5	15.0			35.0	53.5	14.5		288.5 (4)	460 445	1.0 2.5
8	4.0	18.5			7.0	35.5	9.5		95.5		

(1) Most of the hours in these columns are due to unplanned cool downs caused by over-temperature controls turning off heaters.

(2) A new door seal assembly (all polyimide laminate coated with polyphenylquinoxaline) was installed prior to beginning this cycle.

(3) These hours are included in hours reported under 426 - 441°F.

(4) Lower half of tank may have been in the range of 401 to 425°F for 50 hours.

(Table Continued on next page)

TABLE 7 (Continued)
ENVIRONMENTAL CONDITIONING OF TANK NO. 3

Environmental Conditioning Cycle No.	Fuel Liquid Exposure, Hours				Fuel Vapor Exposure, Hours						
	Ambient To 139°F	140 To 150°F	Over Temperature: 151 to ↓ °F	Over Temperature: Hours	Below 140°F (1)	140 To 400°F (1)	401 To 441°F	415 To 441°F	426 To 441°F	Over Temperature: 441°F To ↓°F	Over Temperature: Hours (3)
9	5.5	16.0			87.0	39.5	3.5		162.0	444	3.0
10	1.5	19.5				33.0	8.0		128.5		
11	3.0	18.5				8.0	3.5		161.0		
12	5.0	15.0				6.5	.5		135.5		
13	4.0	17.0				6.5	2.0		138.0		
14	7.0	13.5				6.5	2.5		137.5		
15	4.0	16.5				17.5	2.5		101.0		
16	10.0	14.0				6.5	2.0		162.5		
17	8.0	12.0				8.5	3.5		136.5		
18	7.0	17.5				7.5	2.5		106.5		
19	2.5	23.5				8.5	2.5		135.5		
20	8.0	11.0				9.0	2.5		135.0		
21	10.0	11.0			15.0	30.5	6.5		120.0		
22	10.0	10.0				6.5	3.0		167.0		
1 through 22 TOTALS	132.0	317.0	151-156	14.0	256.0	410.8	112.5		3076.8	441-46[illegible]	6.5

(1) Most of the hours in these columns are due to unplanned cool downs caused by over-temperature controls turning heaters off.

(3) These hours are included in hours reported under 426 - 441°F.

TABLE 7 (Continued)

ENVIRONMENTAL CONDITIONING OF TANK NO. 3

Environmental Conditioning Cycle No.	Fuel Liquid Exposure, Hours				Fuel Vapor Exposure, Hours						
	Ambient To 139°F	140 to 150°F	Over Temperature		Below 140°F (1)	140 to 400°F (1)	401 to 425°F	415 to 441°F	426 to 441°F	Over Temperature	
			151 to ↓ °F	Hours						441°F to↓°F	Hours (3)
23	11.0	8.5			42.0	49.0	7.5		192.0		
24	3.0	18.5				58.0	5.0		108.0		
25	3.0	17.0				5.5	3.0		139.5		
26	3.0	18.0				7.0	2.0		115.0		
27	5.0	15.5				29.0	4.5		257.5		
28	3.0	16.0				39.0	4.0		120.0		
29	17.0	5.0				65.0	43.0	63.5	96.5	446	3.5
30	2.0	17.0				14.0	2.0		105.0		
31	5.0	18.0				2.0	2.0		164.5		
32	1.0		205 175	3.0 19.0		4.0	1.5		141.5		
33	1.0	6.0	175	14.0		9.5	1.5		130.0		
34	2.0	18.5				5.0	1.5		141.0		
35	2.0	8.0	160	10.0		5.0	2.0		141.5		
36	2.0	5.5	170	12.0		55.5	3.5	15.0	196.0		
37	1.0	10.0	170	13.0		50.5	7.0		118.5		
38	2.0	8.0	160	10.0		4.5	2.0		112.5		

(1) Most of the hours in these columns are due to unplanned cool downs caused by over-temperature controls turning heaters off.

(3) These hours are included in hours reported under 426 - 441°F.

TABLE 7 (Continued)
ENVIRONMENTAL CONDITIONING OF TANK NO. 3

Environmental Conditioning Cycle No.	Fuel Liquid Exposure, Hours				Fuel Vapor Exposure, Hours						
	Ambient To 139°F	140 to 150°F	Over Temperature		Below 140°F (1)	140 to 400°F (1)	401 to 425°F	415 to 441°F	426 to 441°F	Over Temperature	
			151 to + °F	Hours						441°F to + °F	Hours (3)
39		2.0	190	18.2		19.5	1.5		171.0		
40	5.0	17.5				8.0	5.5		135.0		
41	1.5	19.5				30.0	5.0		123.0		
42	1.0	24.0				5.0	1.5		131.5		
43	3.0	18.5				8.0	1.5		137.0		
44	1.5	23.5			29.5	12.5	0.3		85.0	448	14
45	1.5	19.5			23.0	28.5	1.5		87.0		
46	1.5	17.0				42.0	2.5		93.5		
47	3.0	22.0			(no vapor exposure due to excessive liquid leakage - see Section 3.8)						
48	11.0	8.5				3.0	1.5		144.5	445	41
49	5.5	12.5				9.5	3.0		138.5		
50	11.5	11.0				10.5	3.0		133.0		
51	14.0	8.5				10.5	2.5		131.5		
52	11.0	12.5				33.5	3.5		163.5		
53	13.0	10.0			1.5	26.5	4.0		121.0		

(1) Most of the hours in these columns are due to unplanned cool downs caused by over-temperature controls turning heaters off.

(3) These hours are included in hours reported under 426 - 441°F.

TABLE 7 (Continued)
ENVIRONMENTAL CONDITIONING OF TANK NO. 3

Environmental Conditioning Cycle No.	Fuel Liquid Exposure, Hours				Fuel Vapor Exposure, Hours						
	Ambient To 139°F	140 to 150°F	Over Temperature		Below 140°F (1)	140 to 400°F (1)	401 to 425°F	415 to 441°F	426 to 441°F	Over Temperature	
			151 to ↓ °F	Hours						441°F to ↓ °F	Hours (3)
54	8.5	14.5				38.5	3.5		87.5		
55	14.0	10.5				3.5	3.0		137.5		
56	9.0	14.0				10.0	2.5		156.5		
57	8.0	15.0				9.0	3.5		133.0		
58	6.0	18.5				9.0	3.0		133.5		
59	10.5	13.0				10.0	2.0		132.5		
60	7.0	17.5				9.5	1.5		131.5		
61	9.5	14.0				9.0	2.5		132.0		
62	11.0	12.5				9.0	2.0		132.5		
63	9.0	15.0				9.5	1.5		141.0		
64	8.5	15.5				9.5	1.5		133.5		
65	8.5	15.0				10.5	2.0		130.0		
66	6.0	19.0				9.0	2.5		131.5		
67	9.0	15.0				10.0	1.0		133.0		
68	8.5	16.5				8.5	2.0		132.5		

(1) Most of the hours in these columns are due to unplanned cool downs caused by over-temperature controls turning heaters off.

(3) These hours are included in hours reported under 426 - 441°F.

TABLE 7 (Continued)
ENVIRONMENTAL CONDITIONING OF TANK NO. 3

Environmental Conditioning Cycle No.	Fuel Liquid Exposure, Hours				Fuel Vapor Exposure, Hours						
	Ambient To 139°F	140 to 150°F	Over Temperature		Below 140°F (1)	140 to 400°F (1)	401 to 425°F	415 to 441°F	426 to 441°F	Over Temperature	
			151 to ↓ °F	Hours						441°F to ↓ °F	Hours (3)
69	9.0	14.0				6.0	2.5		158.5		
70	3.0	16.5				14.0	2.5		115.0		
71	10.5	14.5				8.5	2.5		120.0		
72	2.0	23.5				6.5	3.0		181.0		
73	2.0	21.0				7.0	2.5		81.0		
74	2.0	40.5				22.0	2.0		114.0		
75	16.0	7.5				56.5	5.0		32.5		
76	2.5	22.5				7.0	2.5		135.0		
77	2.0	40.5				6.5	2.0		111.5		
78	1.5	22.0			15.5	14.0	3.0		127.0		
79	7.0	18.5				6.5	2.5		114.0		
80	2.0	22.5			21.5	15.0	3.0		98.5		
81	3.5	18.0				6.5	2.0		137.0		
82	3.0	18.5			71.5	6.5	2.5		62.5		
83	3.5	19.0				6.0	2.0		131.5		

(1) Most of the hours in these columns are due to unplanned cool downs caused by over-temperature controls turning heaters off.

(3) These hours are included in hours reported under 426 - 441°F.

TABLE 7 (Continued)
ENVIRONMENTAL CONDITIONING OF TANK NO. 3

E

Environmental Conditioning Cycle No.	Fuel Liquid Exposure, Hours				Fuel Vapor Exposure, Hours						
	Ambient To 139°F	140 to 150°F	Over Temperature 151 to ↓ °F	Over Temperature Hours	Below 140°F (1)	140 to 400°F (1)	401 to 425°F	415 to 441°F	426 to 441°F	Over Temperature 441°F to ↓ °F	Over Temperature Hours (3)
84	3.0	19.5				5.5	1.5		137.5		
85	3.0	18.5				7.0	2.5		136.0		
86	3.0	19.5				6.0	2.0		138.5		
87	3.0	19.0				6.5	2.0		137.0		
88	3.0	18.0				6.5	2.0		137.5		
89	3.5	19.0				6.5	2.5		136.0		
90	3.0	19.5				6.5	2.5		137.5		
91	3.0	18.5				16.5	2.0		145.5		
92	8.0	14.5				6.0	3.0		138.5		
93	11.5	12.5				6.5	3.5		110.0		
94	3.5	16.5				10.5	2.5		128.5		
95	3.5	19.5				6.5	3.0		132.0		
96	4.5	18.0				7.0	3.5		138.5		
97	3.5	21.0				8.0	3.0		137.5		
98	4.0	19.5				6.5	3.5		129.0		

(1) Most of the hours in these columns are due to unplanned cool downs caused by over-temperature controls turning heaters off.

(3) These hours are included in hours reported under 426 - 441°F.

TABLE 7 (Continued)
ENVIRONMENTAL CONDITIONING OF TANK NO. 3

E

Environmental Conditioning Cycle No.	Fuel Liquid Exposure, Hours				Fuel Vapor Exposure, Hours						
	Ambient To 139°F	140 to 150°F	Over Temperature		Below 140°F (1)	140 to 400°F (1)	401 to 425°F	415 to 441°F	426 to 441°F	Over Temperature	
			151 to ↓ °F	Hours						441°F to↓°F	Hours (3)
99	3.5	18.5				37.5	4.5		138.0		
100	4.0	19.5				54.5	3.0		63.0		
101	3.0	19.0				14.5	2.2		97.5		
102	3.5	21.5				5.5	3.0		114.5		
103	3.0	16.5				6.0	3.5		137.5		
104	3.5	19.5				6.5	3.5		131.0		
105	3.0	20.5				5.5	4.0		132.5		
106	4.0	19.0				5.5	3.0		131.5		
107	3.0	19.5				6.5	3.0		132.5		
108	4.0	19.0				5.5	4.0		133.5		
109	4.5	20.5				14.5	6.5		116.0		
110	4.0	21.5				5.0	3.5		130.5		
111	4.5	20.0				5.5	3.5		131.0		
112	4.5	19.0				6.5	3.5		132.0		
113	5.0	18.0				5.0	3.5		131.5		

(1) Most of the hours in these columns are due to unplanned cool downs caused by over-temperature controls turning heaters off.

(3) These hours are included in hours reported under 426 - 441°F.

TABLE 7 (Continued)
ENVIRONMENTAL CONDITIONING OF TANK NO. 3

E

Environmental Conditioning Cycle No.	Fuel Liquid Exposure, Hours				Fuel Vapor Exposure, Hours						
	Ambient To 139°F	140 to 150°F	Over Temperature		Below 140°F (1)	140 to 400°F (1)	401 to 425°F	415 to 441°F	426 to 441°F	Over Temperature	
			151 to ↓ °F	Hours						441°F to↓°F	Hours (3)
114	4.0	18.5			61	5.0	4.0		44.5		
115	5.5	17.5				6.0	3.5		130.0		
116	4.5	18.5				5.0	4.5		131.5		
117	4.0	18.5				8.5	3.5		27.0		
118	6.0	16.5				5.5	4.5		129.0		
119	4.5	18.5				5.0	3.5		131.0		
120	4.5	19.0				---	---		-----		
121	6.5	17.0				5.0	4.0		139.5		
122	4.0	17.5				5.0	3.5		130.5		
123	4.5	18.5				4.5	3.5		131.0		
124	4.0	18.0				4.5	4.0		132.0		
125	4.5	18.5				12.5	7.0		121.5		
126	4.5	18.0				4.5	4.0		131.0		
127	4.0	19.0				5.5	3.5		129.5		
128	4.5	19.0				4.5	4.5		131.5		

(1) Most of the hours in these columns are due to unplanned cool downs caused by over-temperature controls turning heaters off.

(3) These hours are included in hours reported under 426 - 441°F.

E

TABLE . (Continued)
ENVIRONMENTAL CONDITIONING OF TANK NO. 3

Environmental Conditioning Cycle No.	Fuel Liquid Exposure, Hours				Fuel Vapor Exposure, Hours						
	Ambient To 139°F	140 to 150°F	Over Temperature		Below 140°F (1)	140 to 400°F (1)	401 to 425°F	415 to 441°F	426 to 441°F	Over Temperature	
			151 to ↓ °F	Hours						441°F to ↓ °F	Hours (3)
129	5.0	19.0				4.5	4.5		132.5		
TOTALS (1-129)	676.0	2133.5	151-205°F	113.2	521.5	1794.8	470.0	78.5	16,481.8	442-460	65.0
SUBTOTALS		2,922.7	FUEL LIQ. HRS.				19,346.6	FUEL VAPOR HRS.			
GRAND TOTAL			22,269.3		TOTAL HOURS EXPOSURE						

(1) Most of the hours in these columns are due to unplanned cool downs caused by over-temperature controls turning heaters off.

(3) These hours are included in hours reported under 426 - 441°F.

TABLE 8

CORNER LEAKAGE HISTORY OF TANK NO. 3

Date	Test Interval Completed	Corners Leaking (1)							
		R-1	R-2	R-3	R-4	F-1	F-2	F-3	F-4
3-71	After 8 Env. Cond. Cycles		X	X	X		X	X	X
8-71	After Repairs of Corners		X					X	
9-71	After Load Cycling at 3 Temperatures		X		X			X	
1-72	After 22 Env. Cond. Cycles		X		X	X		X	X
1-72	After Load Cycling at 3 Temperatures	X	X		X	X		X	X
2-72	After Repairs		X				X	X	X
8-72	After 39 Env. Cond. Cycles Plus Loading at 3 Temperatures	X	X			X		X	
9-72	After Repairs of R-1 & R-2					X		X	
11-72	After 46 Env.Cond.Cycles	X	X	X	X	X	X	X	X
11-72	After Repairs of R-1,R-2,R-3,R-4				X	X	X	X	X
2-73	After 54 Env. Cond. Cycles	X	X	X	X	X	X	X	X
2-73	After Repairs of R-1,R-2,R-3,R-4					X	X	X	X
8-73	After 74 Env.Cond.Cycles		X					X	X
2-74	After 92 Env.Cond.Cycles			X	X	X	X	X	X
7-74	After 117 Env.Cond.Cycles		X		X	X	X	X	X
9-74	After 120 Env.Cond.Cycles (2)		X		X			X	X
11-74	After 129 Env.Cond.Cycles	X	X	X	X	X	X	X	X

(1) See page B-15 for locations of corners.
(2) Corners F-1 and F-2 were repaired.

REV LTR: E

APPENDIX A

LEAKAGE TESTS - TANK NUMBERS 1 AND 2

LEAKAGE TEST DATA SHEETS

LEGEND FOR LEAKAGE SYMBOLS:

(1) SEALANT FILLET OVER FASTENER SHOWS LOSS OF ADHESION TO TITANIUM.

(2) SMALL CRACK IN FILLET OVER FASTENER

(3) INADEQUATE FILLET APPLICATION

(4) CAUSE OF LEAK UNKNOWN

(5) LOSS OF ADHESION, BUT NO LEAK

(6) SEALANT FILLET CRACKED

(7) SEALANT FILLET SHOWS LOSS OF ADHESION TO TITANIUM

(8) SEALANT FILLET SHOWS LOSS OF ADHESION TO SERMETAL W

(9) EVIDENCE OF SEALANT REVERSION

(10) FILLET CRACKED OR LOOSENED BY EXPANSION OF INJECTION UNDERNEATH.

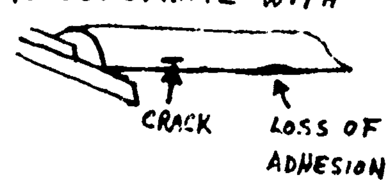
(11) GENERAL OR CONTINUOUS JOINT LEAKAGE

(12) LOSS OF ADHESION OF REPAIR FILLET TO ORIGINAL FILLET

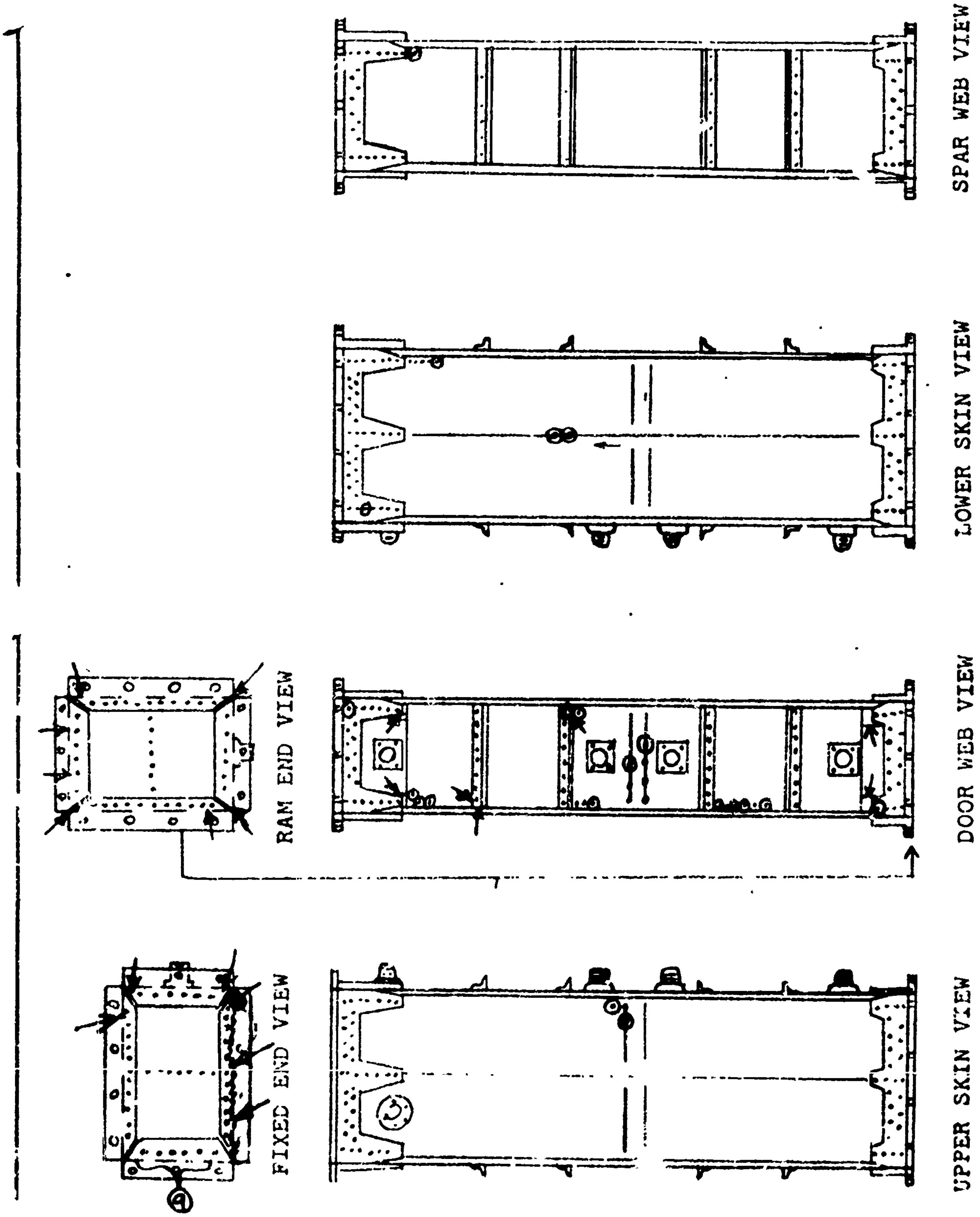
(13) LOSS OF ADHESION OF REPAIR FILLET TO STRUCTURE

(14) CRACK IN REPAIR FILLET

(15) SMALL CRACK OR LOSS OF ADHESION IN JOINT FILLET, GENERALLY $\frac{1}{8}$-$\frac{1}{4}$ INCH LONG. CRACK GENERALLY PENETRATES TO SUBSTRATE WITH SOME ACCOMPANYING LOSS OF ADHESION.

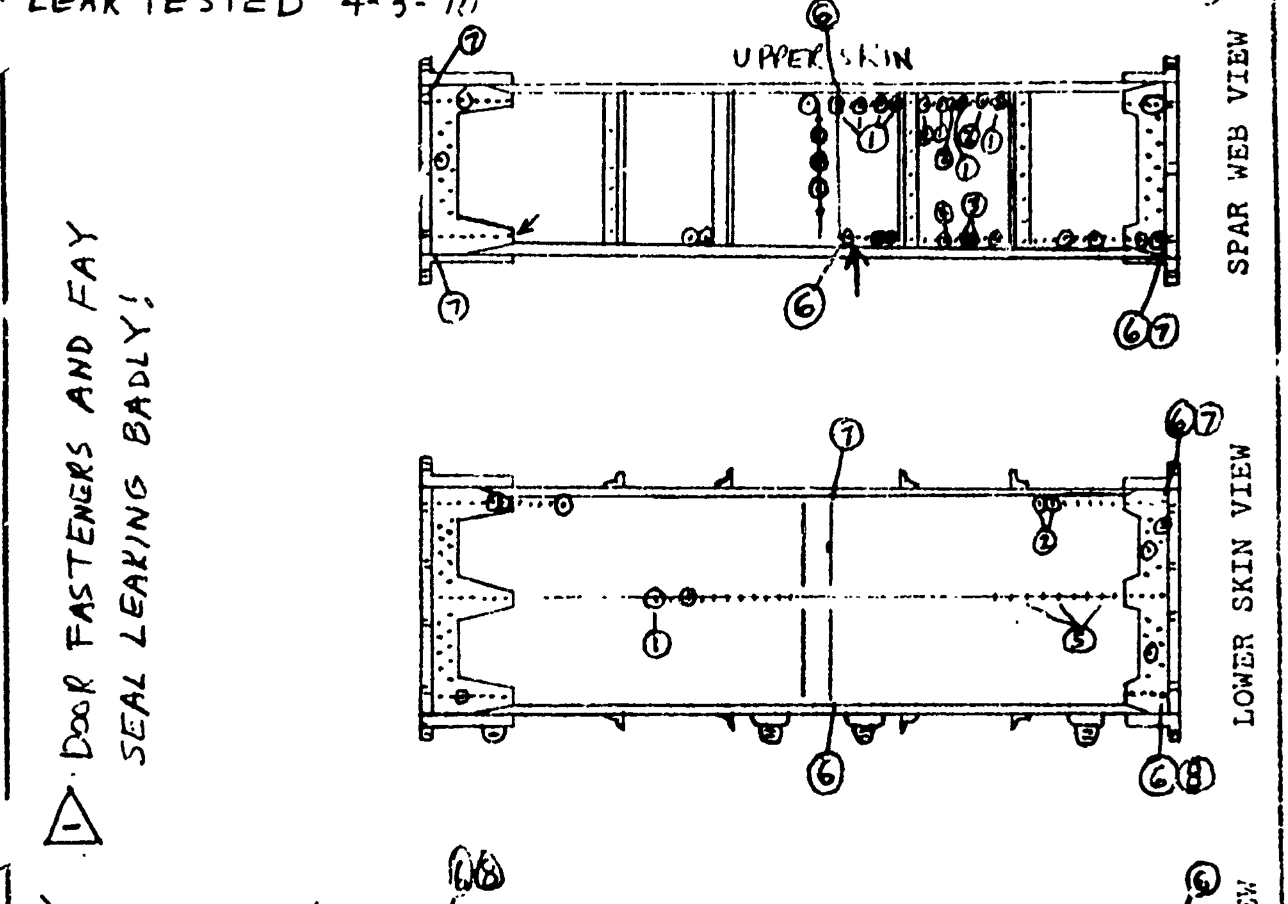


LEAKS MARKED WITH CIRCLES (FASTENER LEAKS) OR ARROWS
LEAK TESTED 3-17-70 (SEAM LEAKS)



TANK NO. 2 - LEAKAGE AFTER 524 HOURS ENV. CONDITIONING AT 415-441°F.

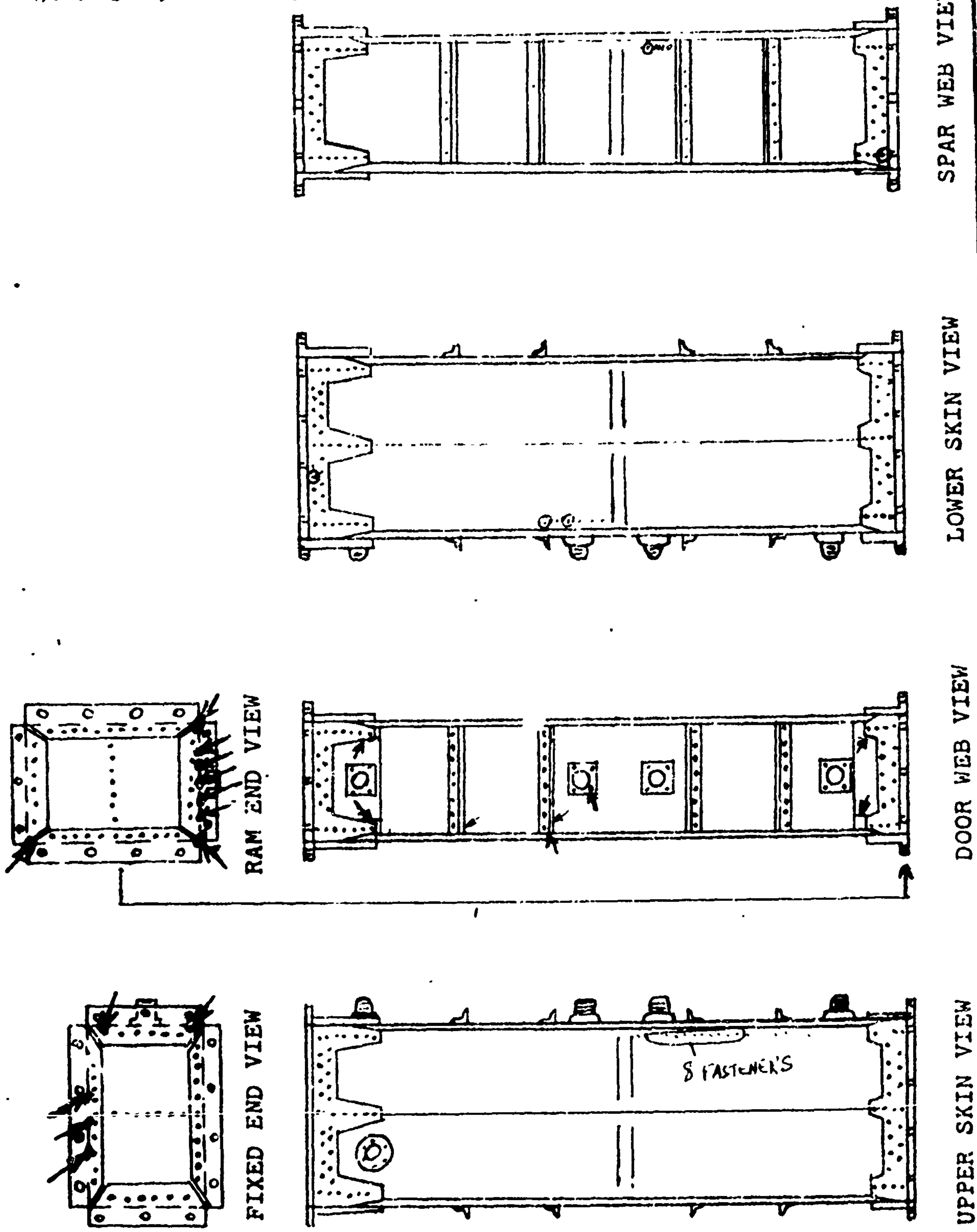
LEAKS MARKED WITH CIRCLES (FASTENER LEAKS) OR ARROWS (SEAM LEAKS)
LEAK TESTED 4-3-70



TANK NO. 2 - LEAKAGE AFTER 524 HOURS ENV. CONDITIONING PLUS 510 LOAD CYCLES AT 426-441°F.

LEAKS MARKED WITH CIRCLES (FASTENER LEAK) OR ARROWS ([illegible]

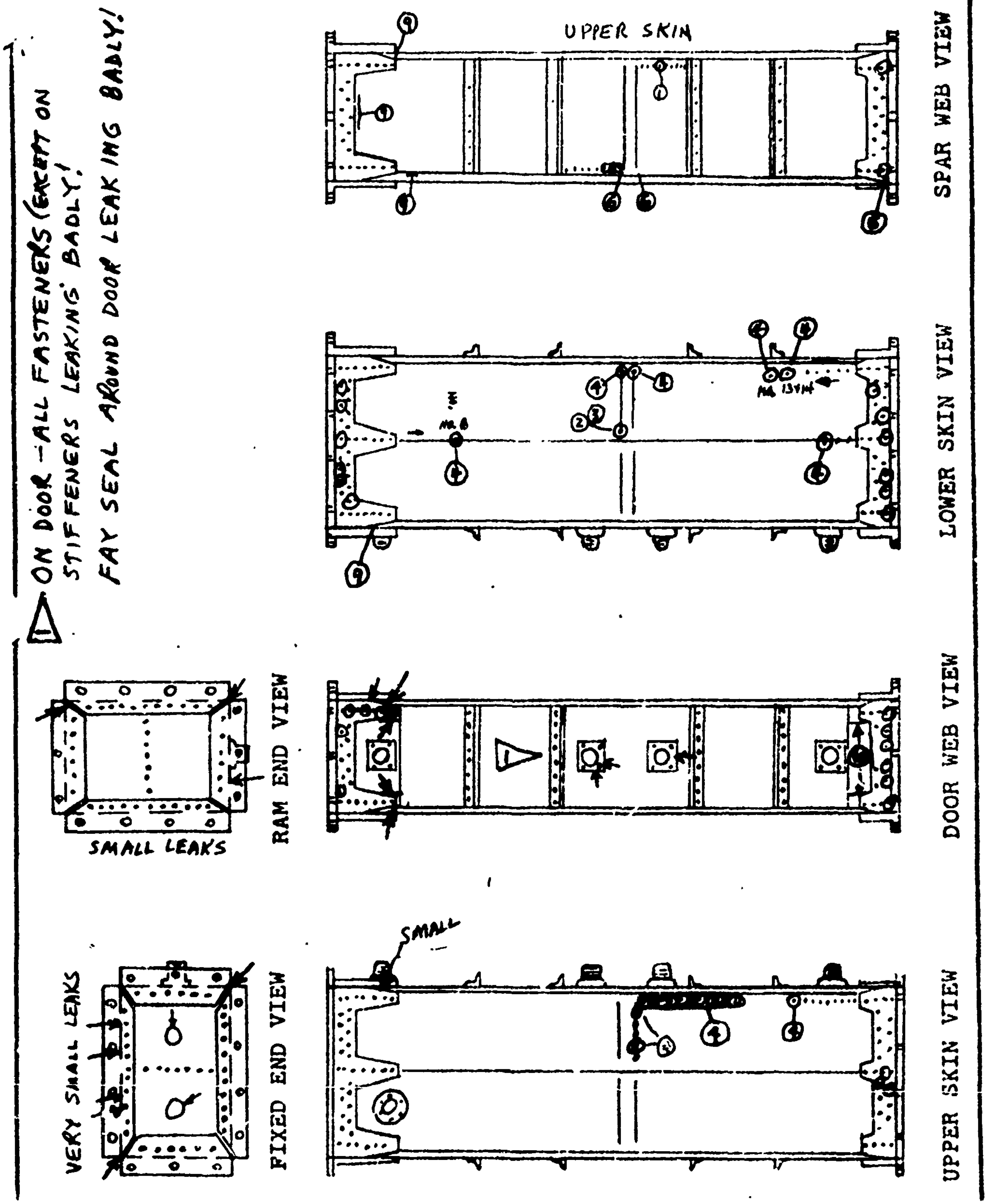
LEAK TESTED 3-17-70



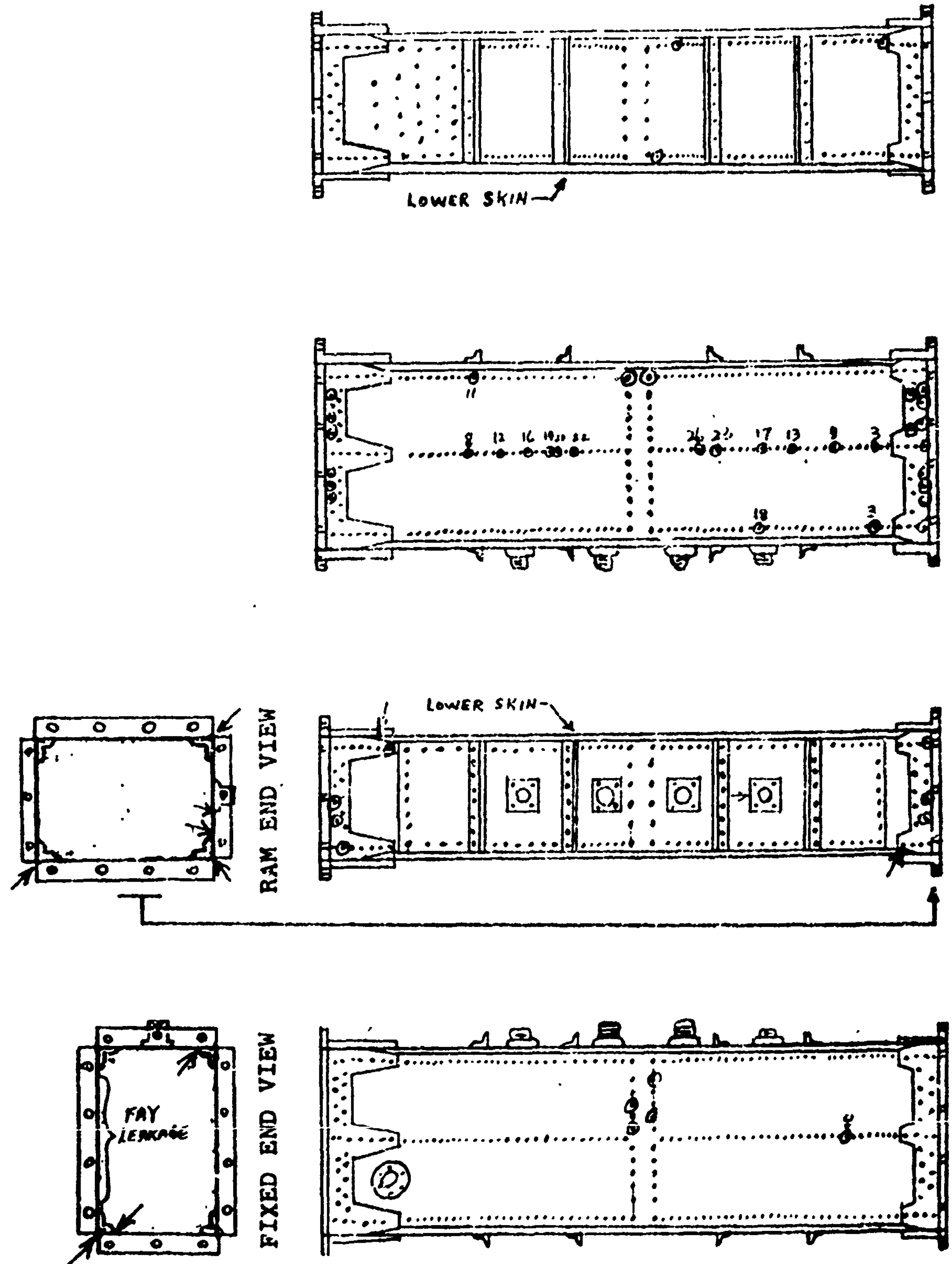
TANK NO. 1 - LEAKAGE AFTER 521 HOURS ENV. CONDITIONING AT 415 441°F. (LEAKAGE TEST NO. 1)

LEAKS MARKED WITH CIRCLES (FASTENER LEAKS) OR ARROWS (SEAM LEAKS)

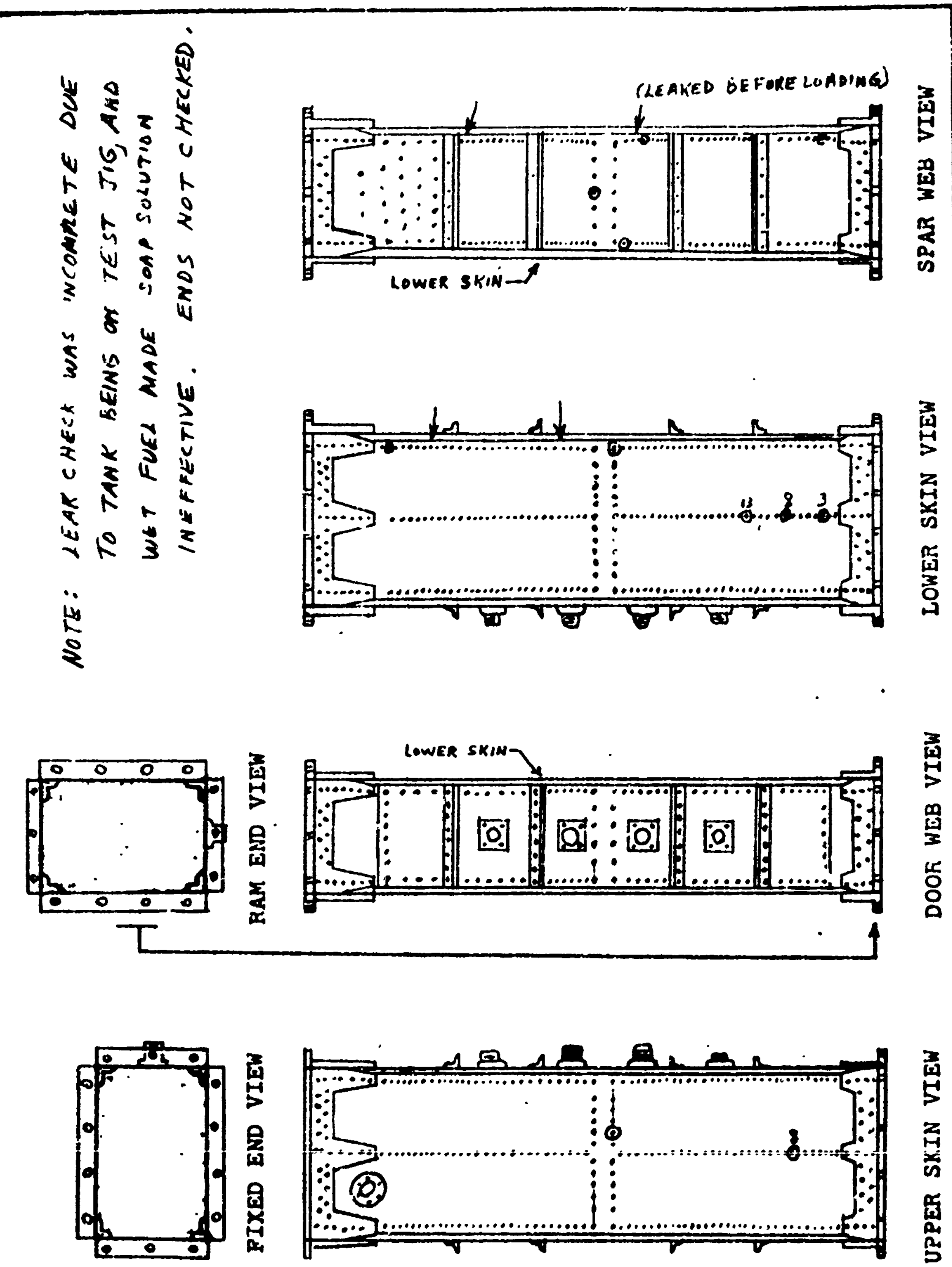
LEAK TESTED 3-31-70



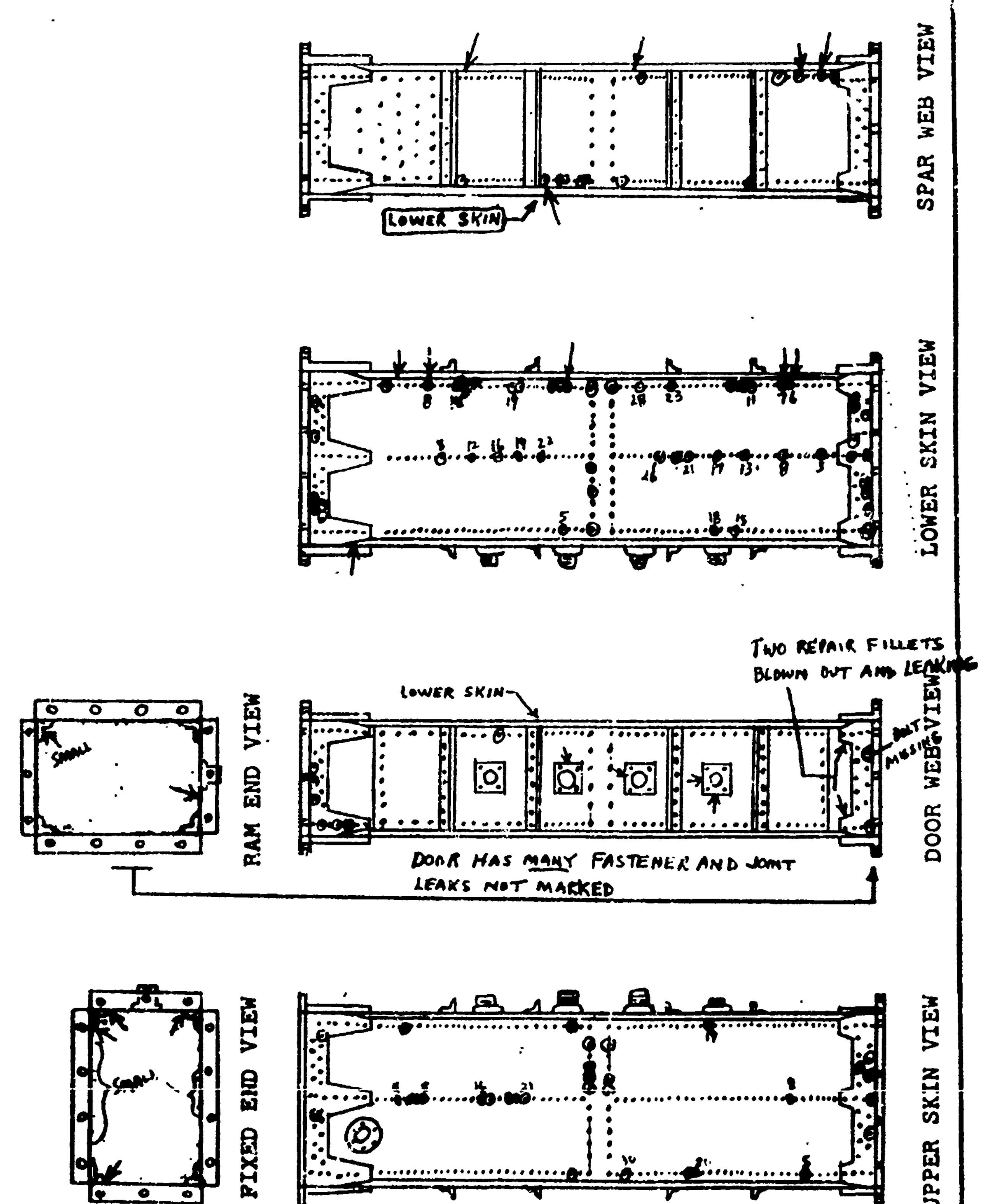
TANK NO. 1 - LEAKAGE AFTER 524 HRS ENV COND. PLUS 510 LOAD CYCLES AT 426-441°F. (LEAKAGE TEST NO. 2) PAGE A-5



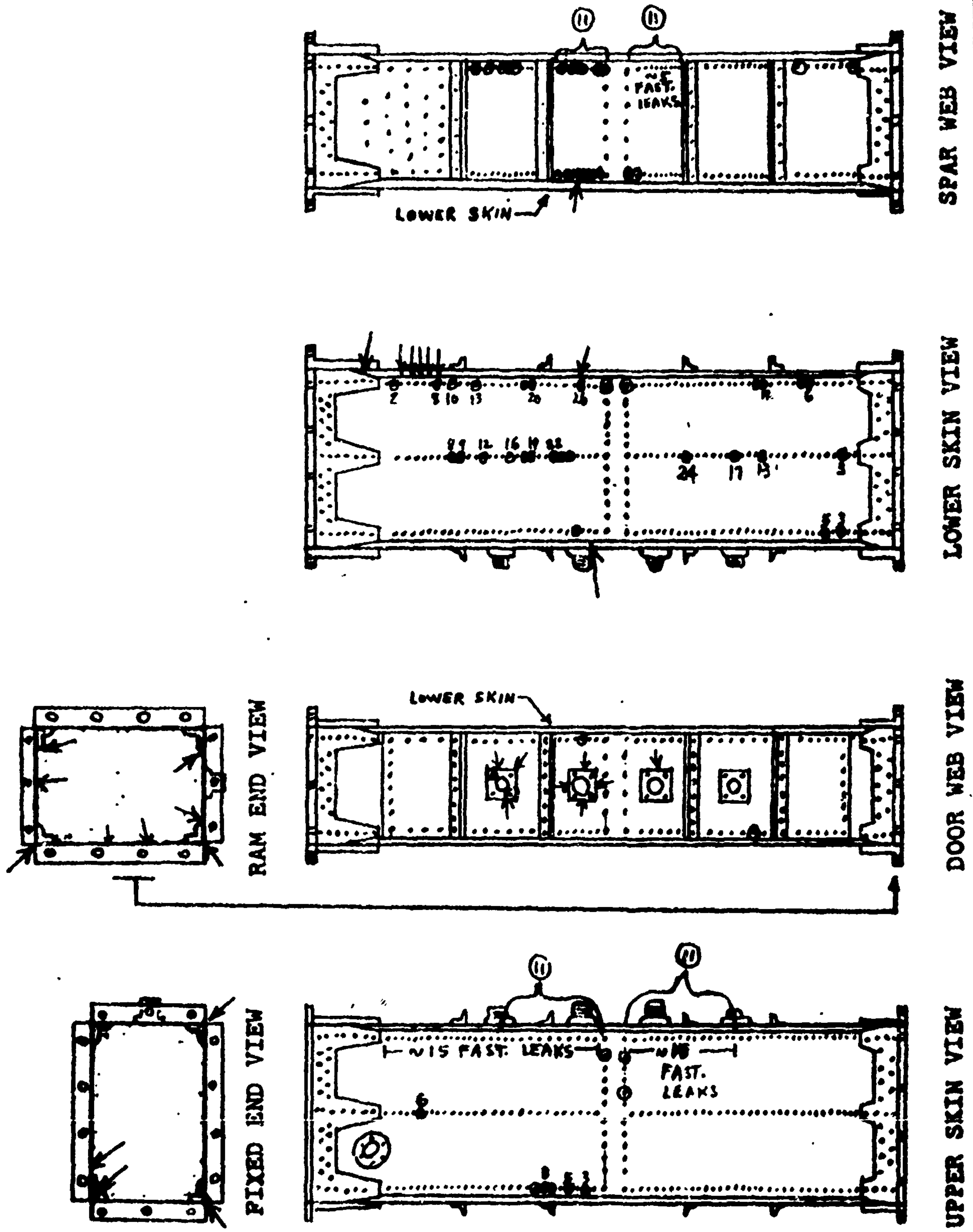
TANK NO. 1 - LEAKAGE TEST NO. 3 (AFTER REPAIRS PER 6.1)



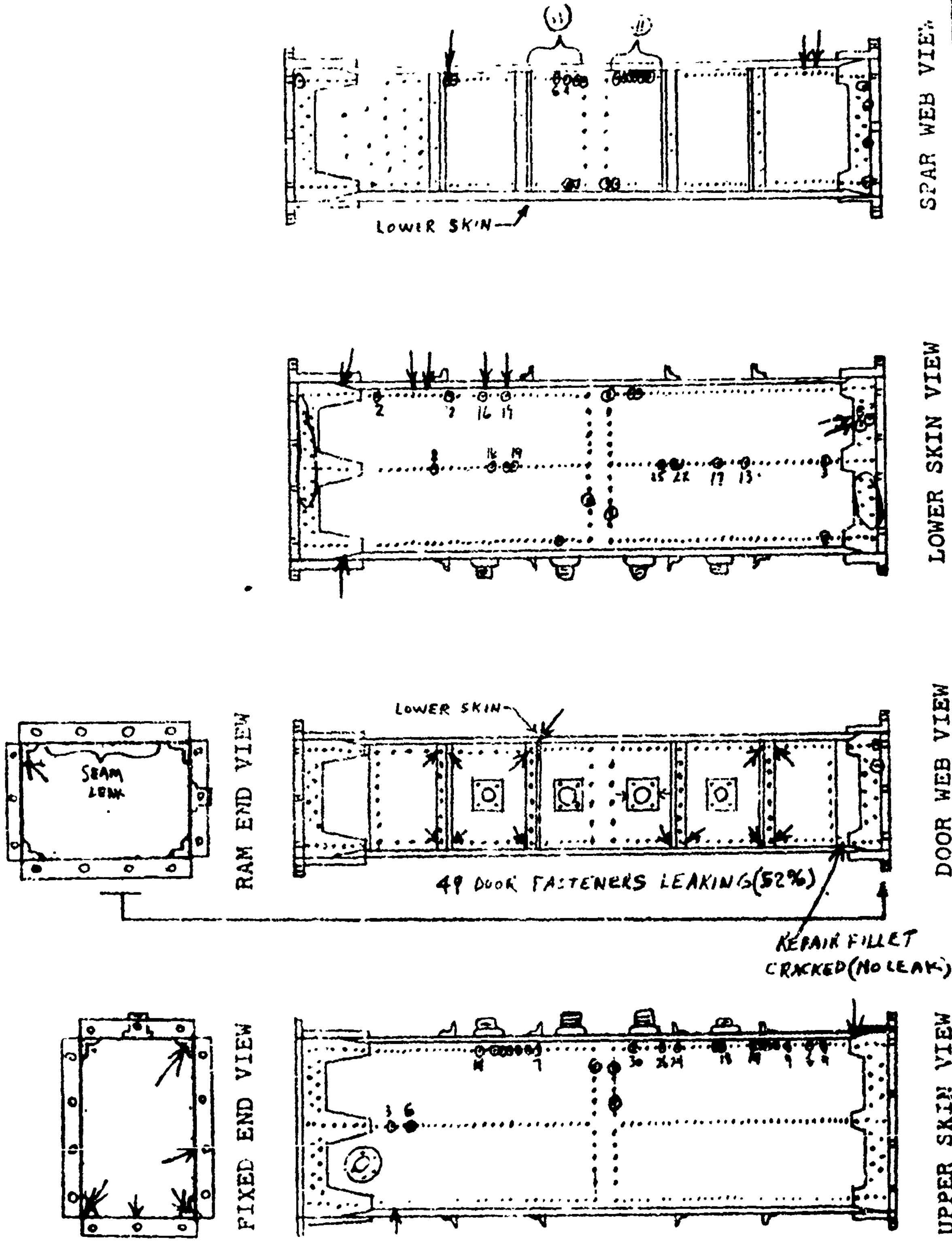
TANK NO. 1 - LEAKAGE TEST NO. 4 (COLD AND ROOM TEMP. CYCLING FOLLOWING REPAIRS PER 6.1)



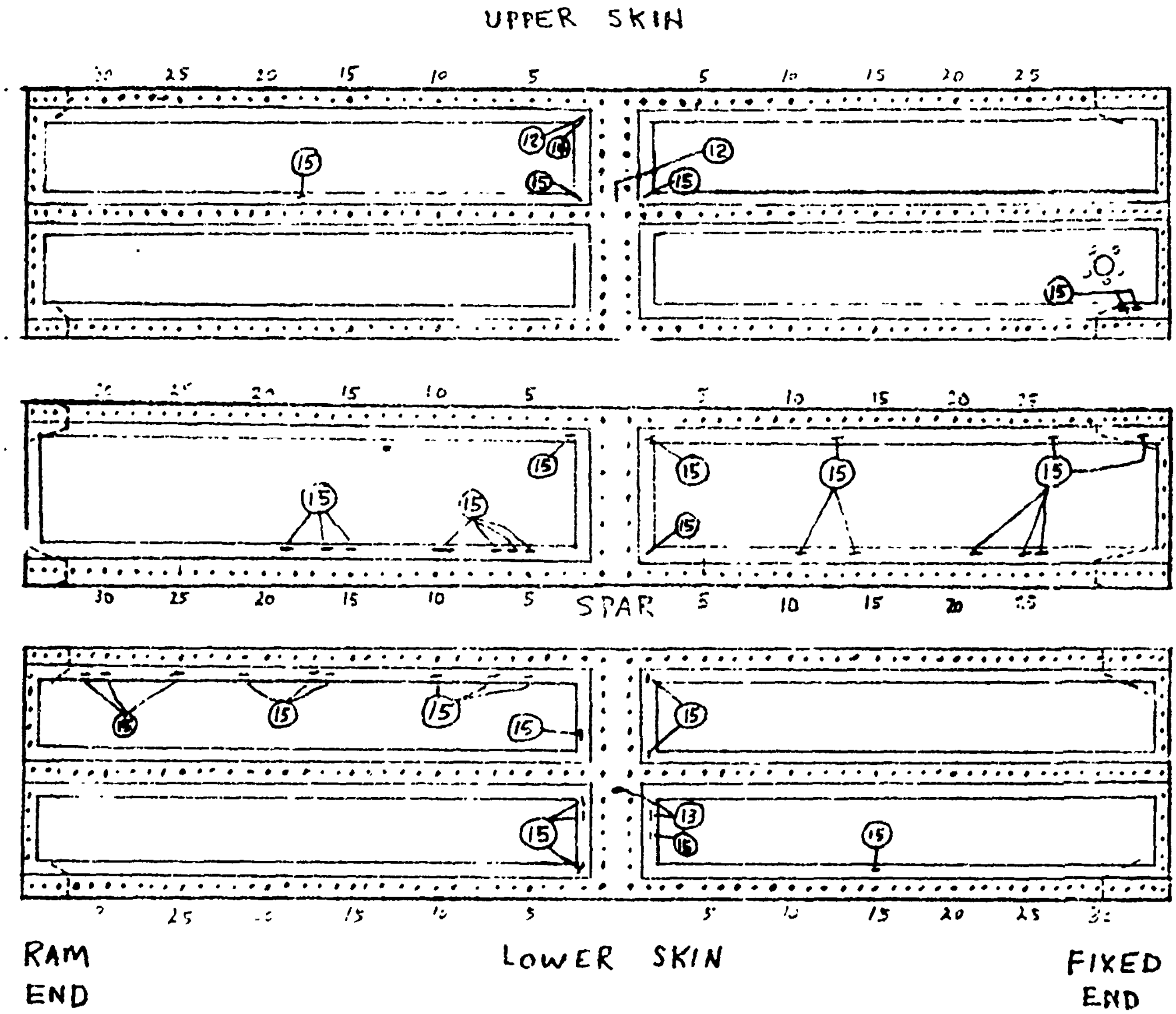
TANK NO. 1 - LEAKAGE TEST NO. 5 (50 LOAD CYCLES HOT, AFTER REPAIRS)



TANK NO. 1 - LEAKAGE TEST NO. 6 (AFTER 1572 HOURS ENV. COND. AT 415-441°F)



TANK NO 1 - LEAKAGE TEST NO. 7 (AFTER HOT, ROOM TEMP., AND COLD DYNAMIC CYCLING)



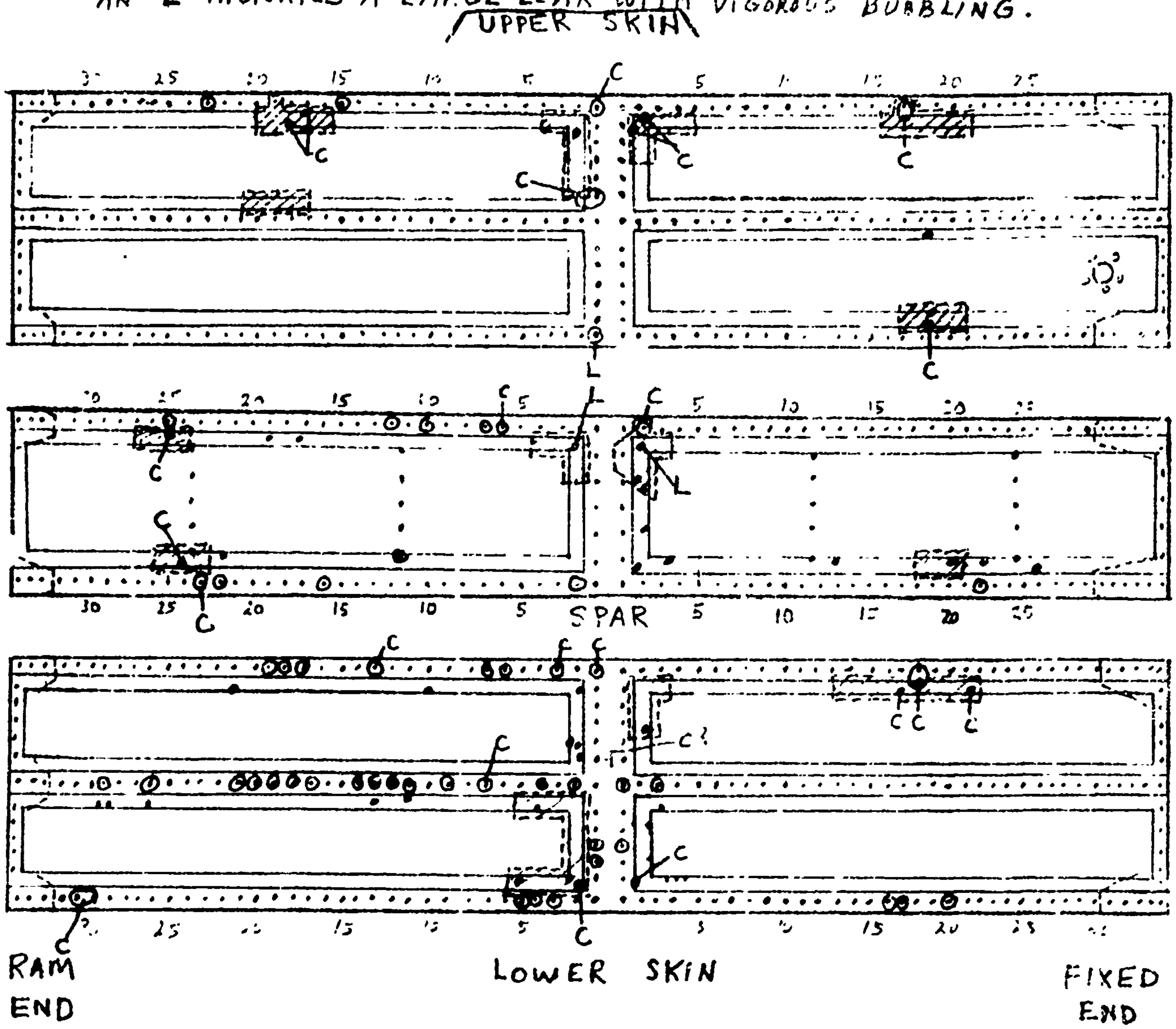
FILLET DEFECTS OBSERVED AFTER LEAKAGE TEST NO.7 (PRIOR TO INTERNAL LEAKAGE ANALYSIS)

TANK NO.

LEGEND:

(FILLET) ——•—— } DOT ON FILLET OR [illegible] FASTENER [illegible]
(FASTENER) ····⊙···· } WHERE BUBBLE FORMED ON SEALANT. NO CONTINUOUS BUBBLING.

A "C" POINTING TO ABOVE AREAS INDICATES A SMALL LEAK WITH CONTINUOUS BUBBLING.
AN "L" INDICATES A LARGE LEAK WITH VIGOROUS BUBBLING.

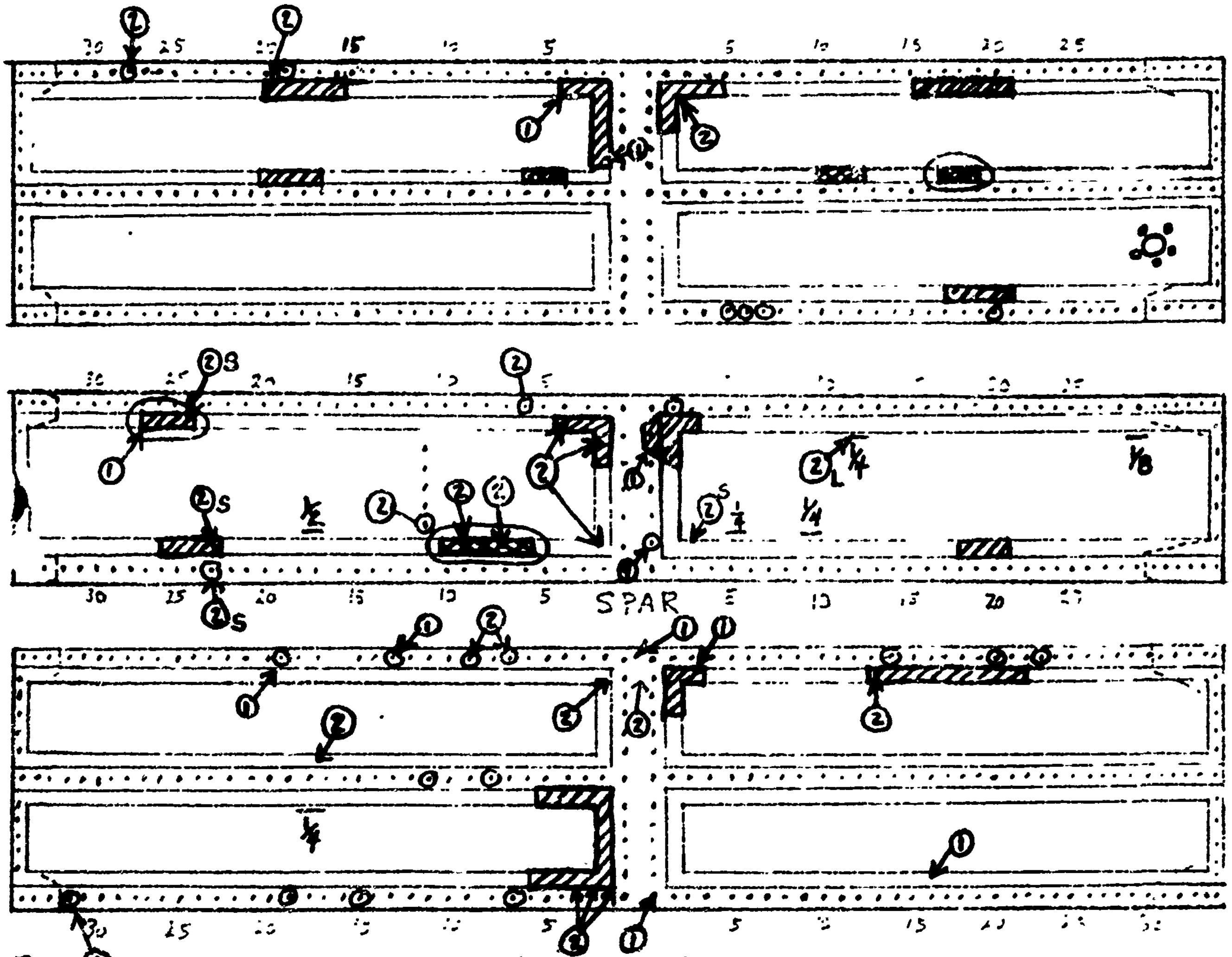


TANK NO. 1 INTERIOR LEAKAGE ANALYSIS CONDUCTED AFTER 12 ENVIRONMENTAL CONDITIONING CYCLES. ANALYSIS CONDUCTED USING 5 PSIG NEGATIVE (VACUUM) PRESSURE IN TANK WITH WATER LAYER ON INTERIOR TO VISUALLY INDICATE LEAKAGE BY BUBBLING.

PAGE A-12

B

UPPER SKIN



⊙ EXISTING LEAKING FASTENERS AFTER 12 ENV. COND. CYCLES (NOT REPAIRED)

FILLET REPAIRED AFTER 12 ENV. COND. CYCLES (AUG 70)

← ① LEAK AFTER 20 ENV. COND. CYCLES (NOV 70)

FILLET REPAIRED AFTER 20 ENV. COND. CYCLES (NOV 70)

← ② LEAK AFTER 31 ENV. COND. CYCLES (AUG 71)

FILLET REPAIRED, ADHESION CHECKED, THEN RE-REPAIRED

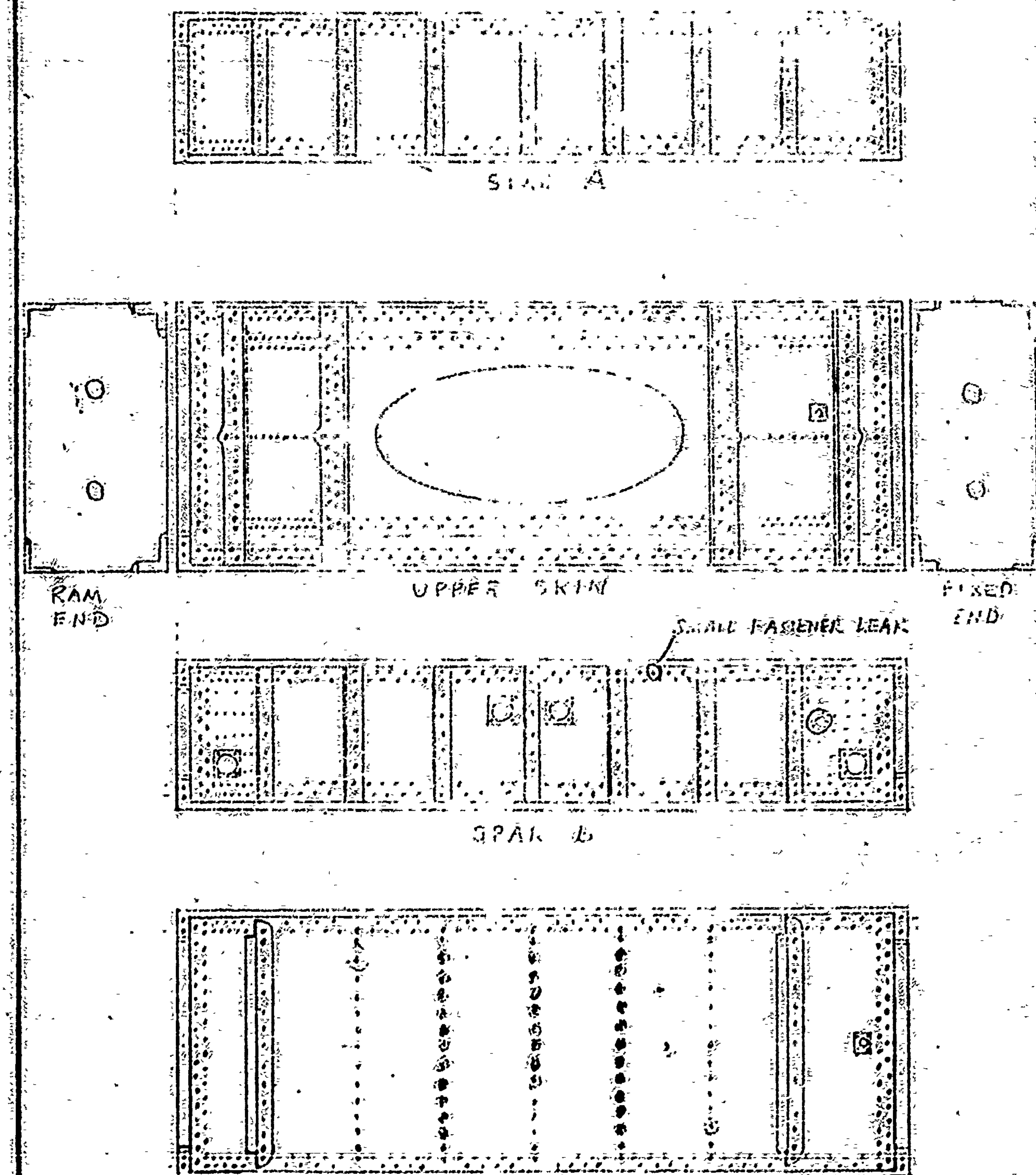
— EXISTING FILLET CRACK AFTER 20 ENV. COND. CYCLES $\frac{1}{X}$ = ~ LENGTH

L = LARGE LEAK
S = SMALL LEAK

APPENDIX B

LEAKAGE TESTS - TANK NUMBER 3

11-6-70



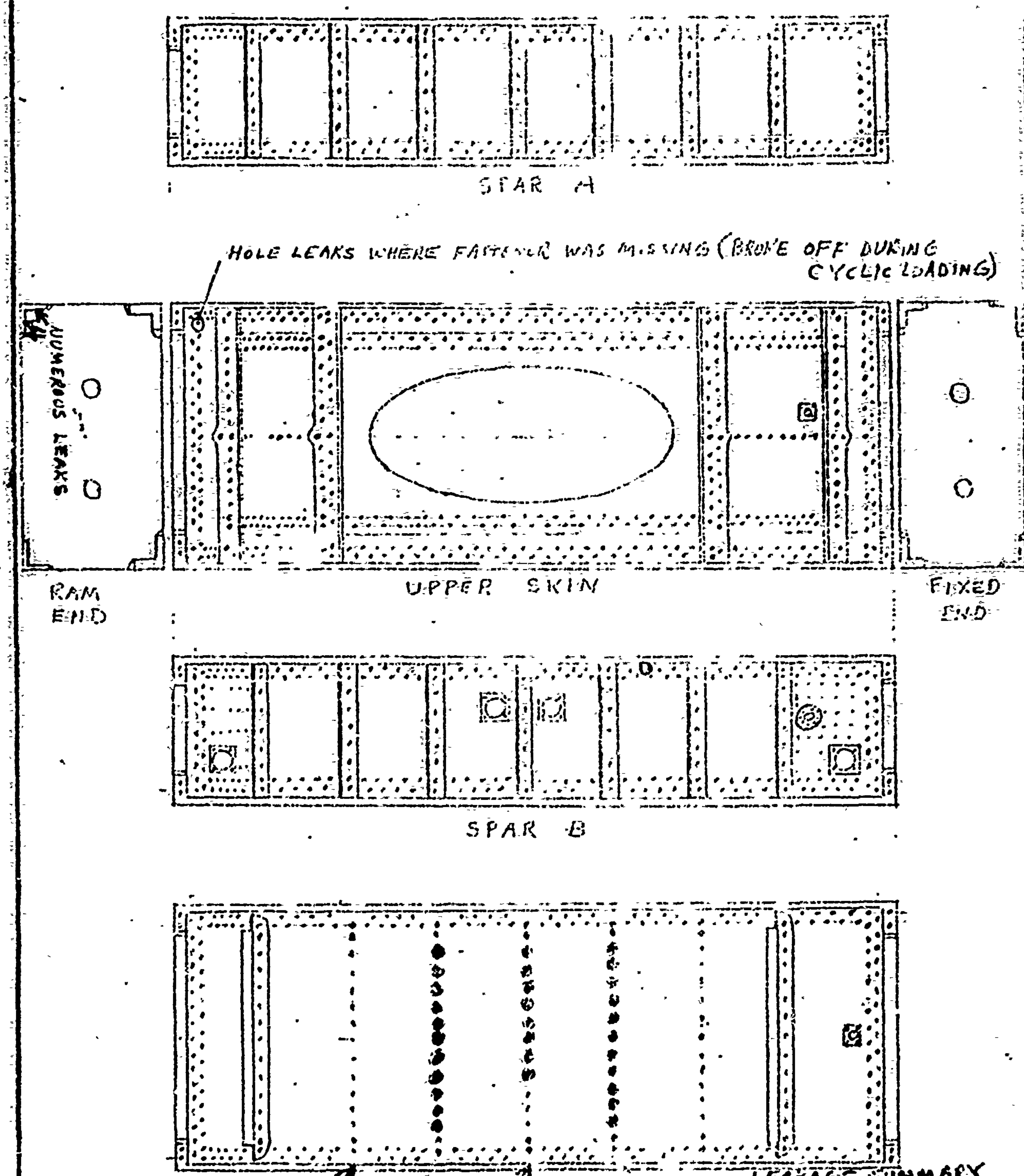
LEAKAGE SUMMARY

FASTENERS	JOINTS	CORNERS
1	0	0

CALC			REVISED	DATE	TANK NO. 3 LEAKAGE TEST NO. 1
CHECK					ORIGINAL LEAK TEST - PRIOR TO ANY TESTING AT WICHITA
APR					
APR					THE BOEING COMPANY
CONTRACT NO.					DATA SHEET NO. 3-1

PAGE B-1

12-4-70

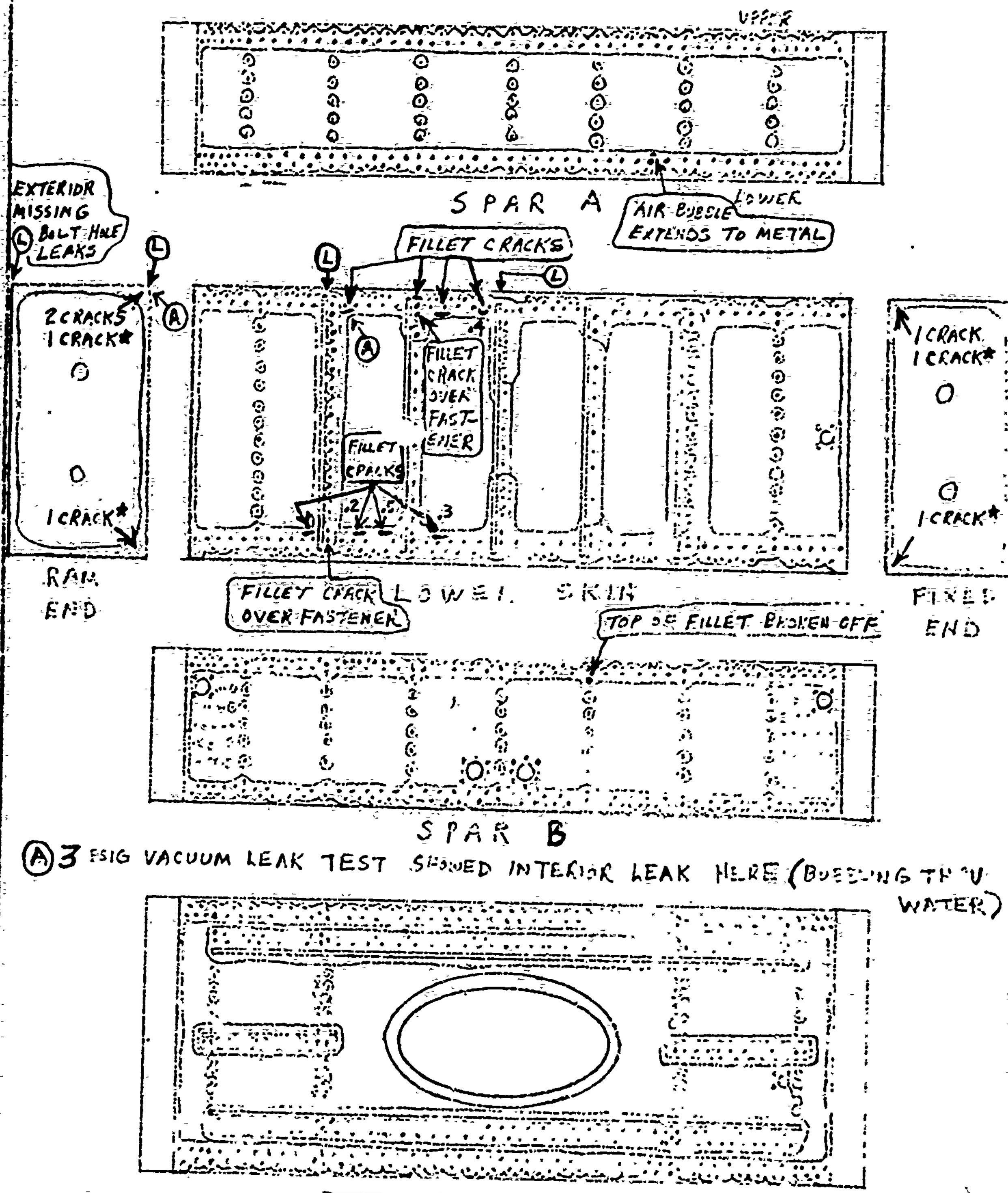


LEAKAGE SUMMARY

FASTENERS	JOINTS	CORNERS
1	2	2

CALC			REVISED	DATE	TANK NO. 3 LEAKAGE TEST NO. 2. LEAK TESTED AFTER INITIAL LOAD CYCLING AT ALL 3 TEMPERATURES
CHECK					
APR					
APR					THE [illegible] COMPANY
					PAGE B-2
CONTRACT NO.					DATA SHEET NO. 3-2

12-4-70



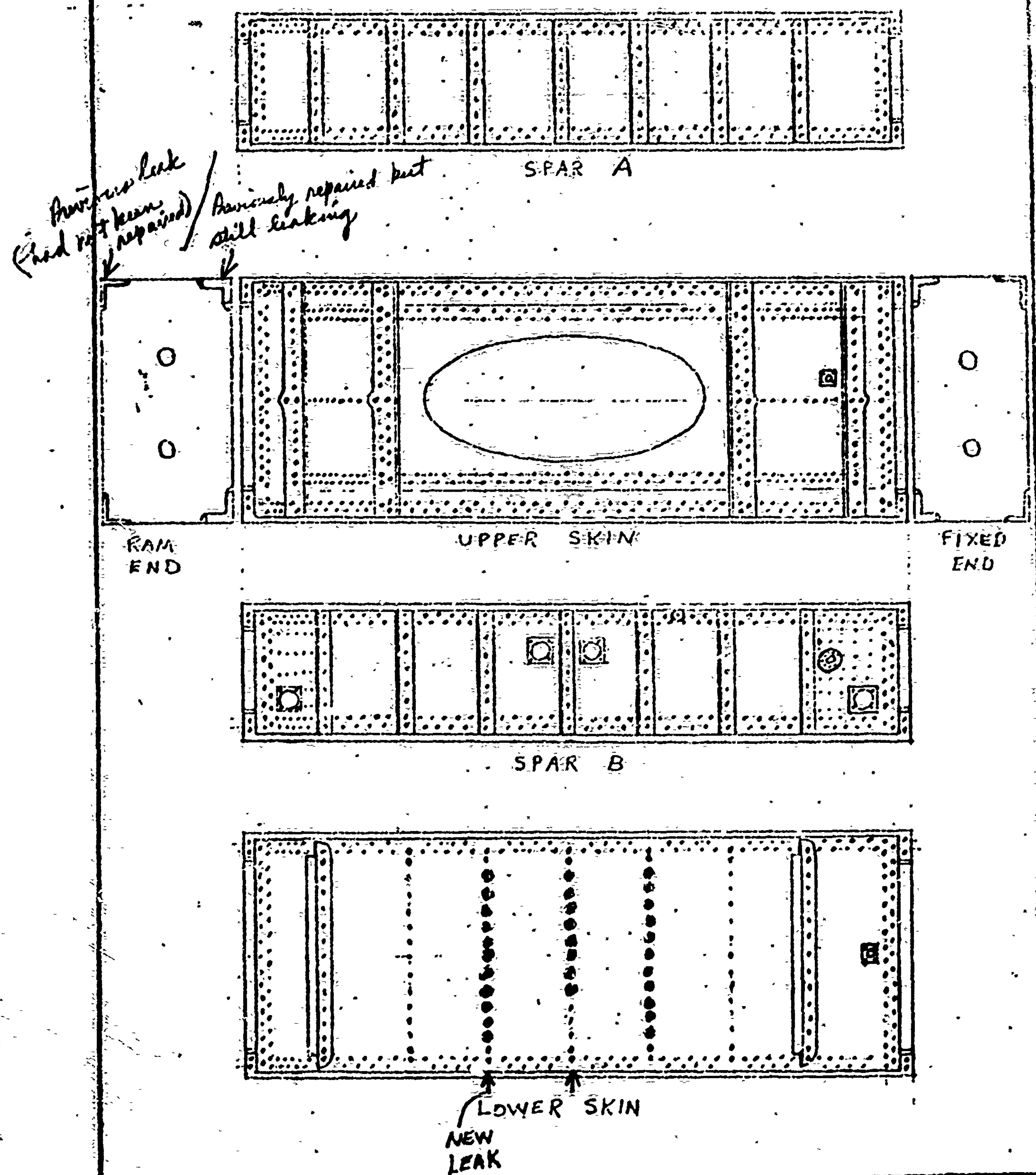
NUMBERS INDICATE CRACK LENGTH

(L) = EXTERIOR JOINT LEAK

★ = MAY BE INCOMPATIBILITY OF REPAIR OVER CURED SEALANT

			REVISED	DATE	TANK NO. 2 INTERIOR VIEW AFTER INITIAL LOAD CYCLING AT 3 TON [illegible]	
CALC						
CHECK						
APR						D3-8297
APR					THE [illegible] COMPANY	
						PAGE
CONTRACT NO.	Rev. E				DATA [illegible] 3	B-3

1-8-71



			REVISED	DATE
CALC				
CHECK				
APR				
APR				

TANK NO. 3 LEAKAGE TEST NO. 3.
LEAK TESTED AFTER 1500 LOAD CYCLES AT R.T. PLUS 500 HOT CYCLES.

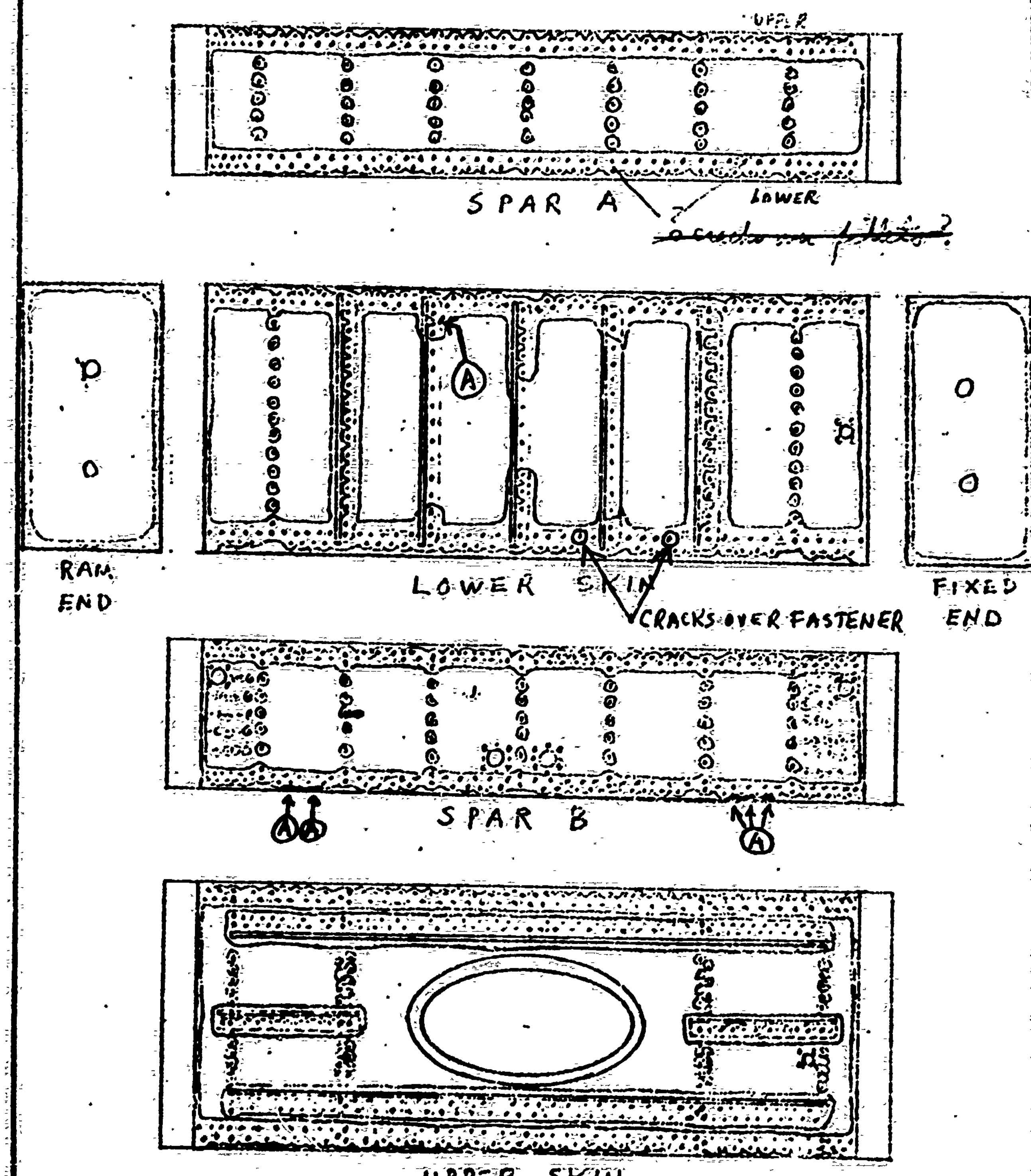
THE BOEING COMPANY

CONTRACT NO.

DATA SHEET NO. 3-4

PAGE B-4

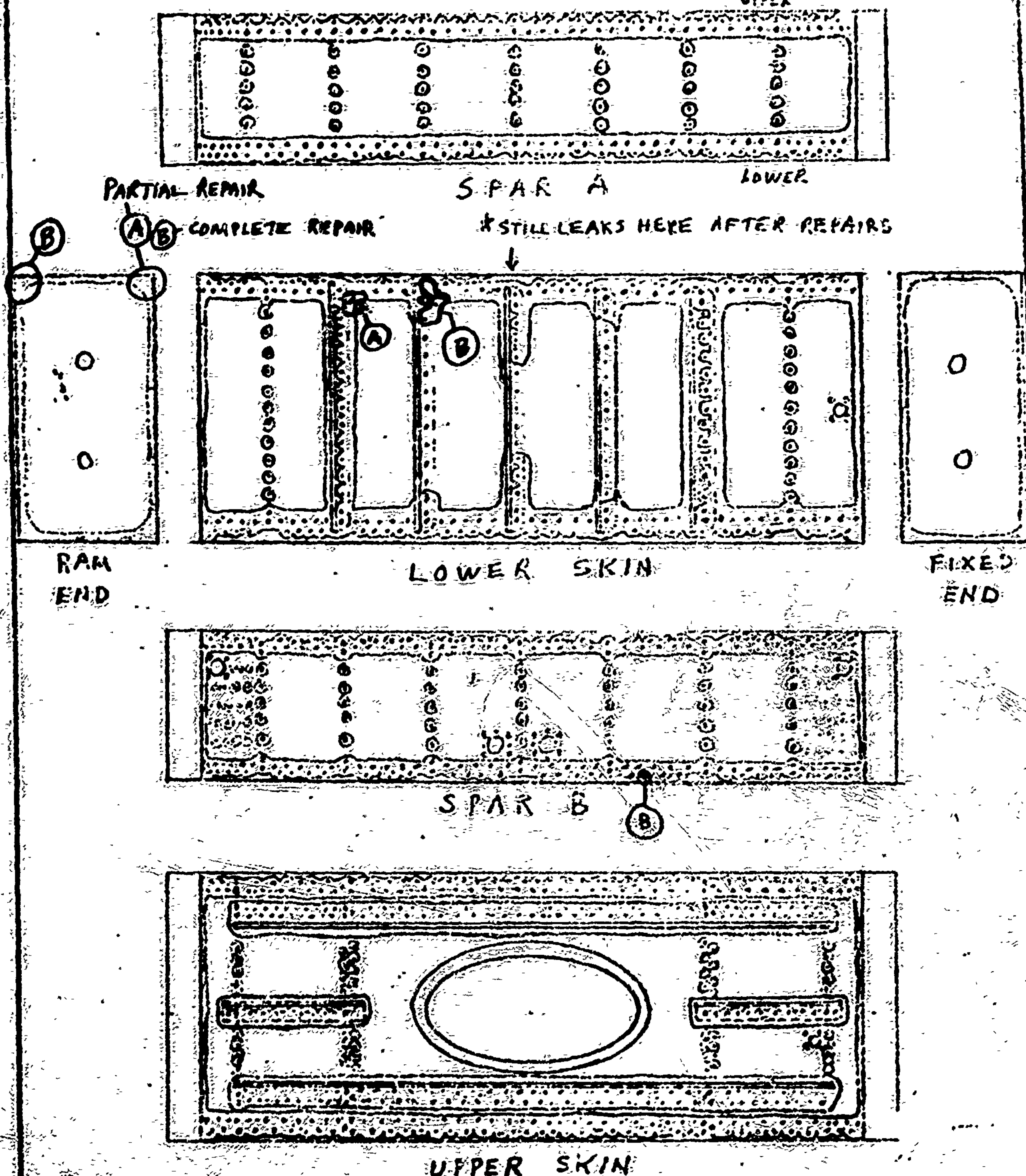
E-282 R4



Ⓐ VERY SMALL FILLET CRACKS

*CRACKS IN ADDITION TO THOSE SHOWN IN DATA SHEET NO. 3-3

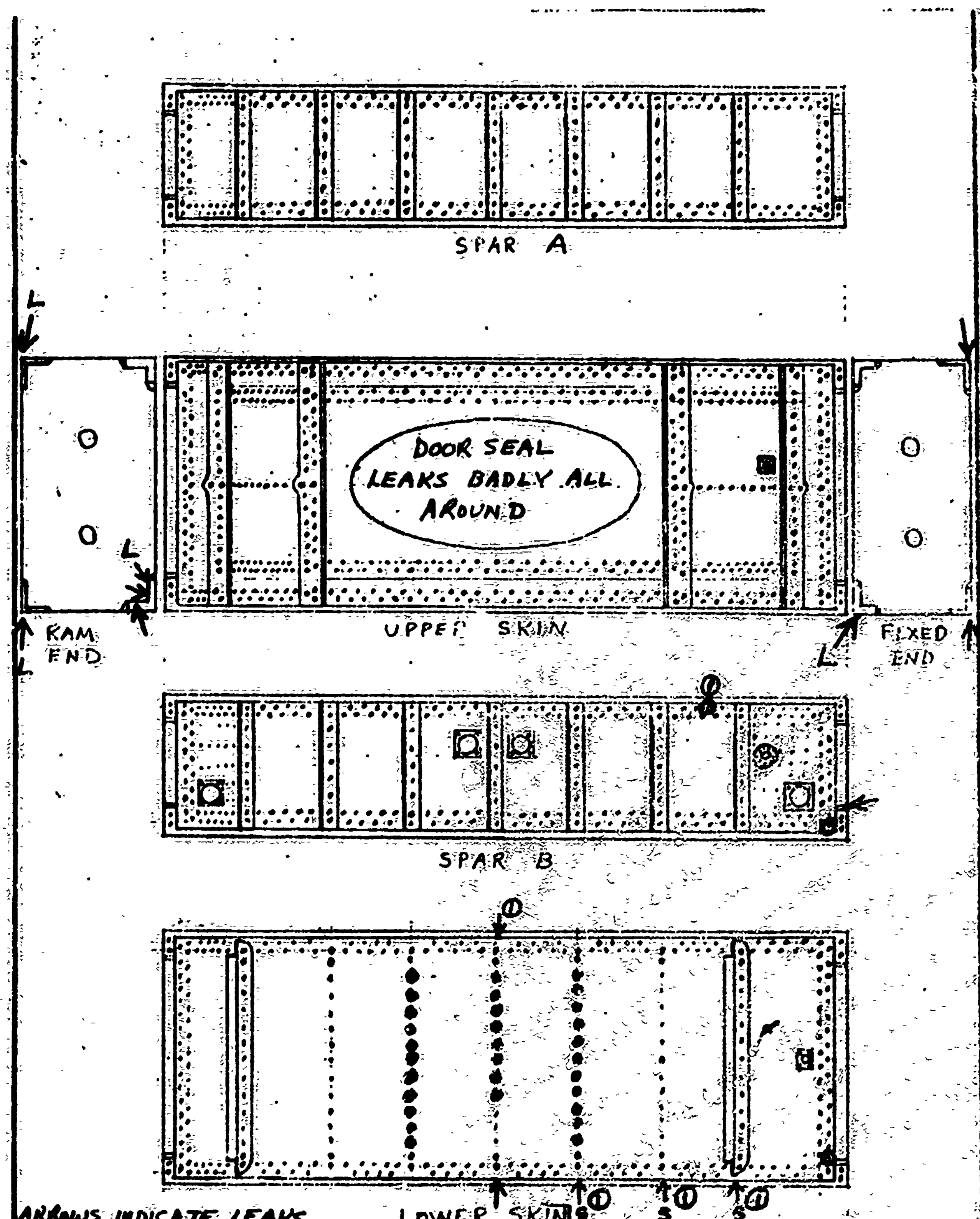
CALC			REVISED	DATE	TANK NO. 3 INTERIOR VIEW SHOWING ADDITIONAL* CRACKS AFTER 1500 R.T. CYCLES + 500 HOT CYCLES	
CHECK						
AP					THE BOEING COMPANY	
APR						PAGE B-5
CONTRACT NO.					DATA SHEET NO. 3-5	



(A) REPAIRED BY JM ON 1-6-71
(B) " " " ON 1-11-71 (THESE REPAIRS STOPPED ALL BUT ONE* LEAK)

CALC			REVISED	DATE	TANK NO. 3 INTERIOR VIEW SHOWING AREAS REPAIRED IN JAN 71	
CHECK						
APD					THE BOEING COMPANY	
APD						PAGE B-6
					DATA SHEET No. 3-6	

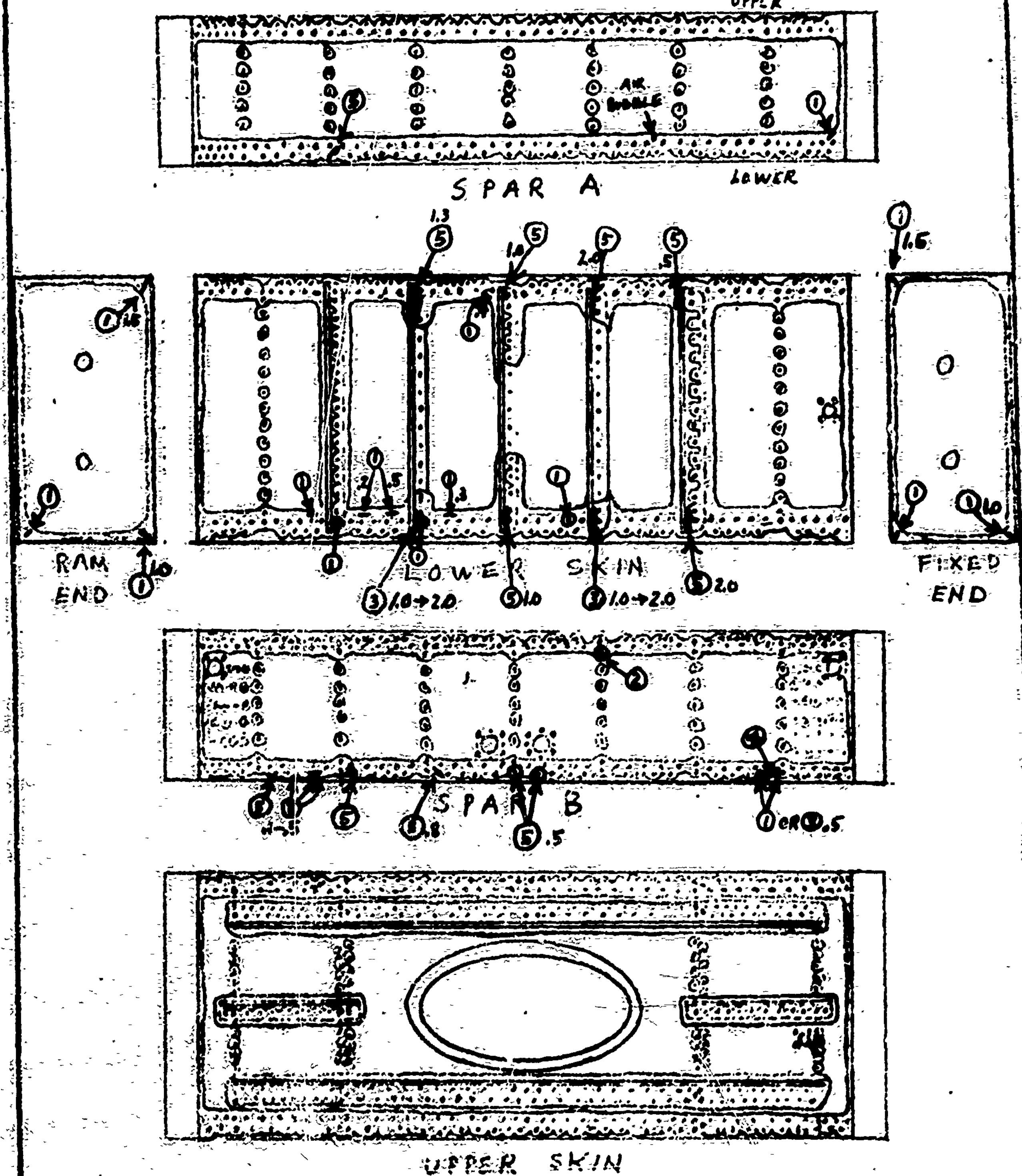
CONTRACT N



ARROWS INDICATE LEAKS
L = LARGE LEAK (RAPID BUBBLING)
S = SMALL LEAK (SLOW BUBBLING)
TEST CONDUCTED 3-31-71

① ADDITIONAL LEAKS DURING SECOND LEAK TEST 8-3-71 (ORIGINAL LEAK TEST WAS 3-31-71)

CALC	LM	8-6-71	REVISED	DATE	TANK NO. 3 LEAKAGE TEST NO. 4 AFTER 8 ENVIRONMENTAL CONDITIONING CYCLES
CHECK					
APR					THE BOEING COMPANY
APR					
					PAGE 8-7
CONTRACT NO.					



SEE PAGE B-8A FOR CODE IDENTIFICATION

TANK INSPECTED 8-6-71

CALC	L.M.	8-6-71	REVISED	DATE
CHECK				
APD				
APD				

TANK NO. 3 INTERIOR VIEW AFTER 8 ENVIRONMENTAL CONDITIONING CYCLES

THE [illegible] COMPANY

PAGE B-8

CONTRACT NO.

B

CODE FOR PAGE B-8

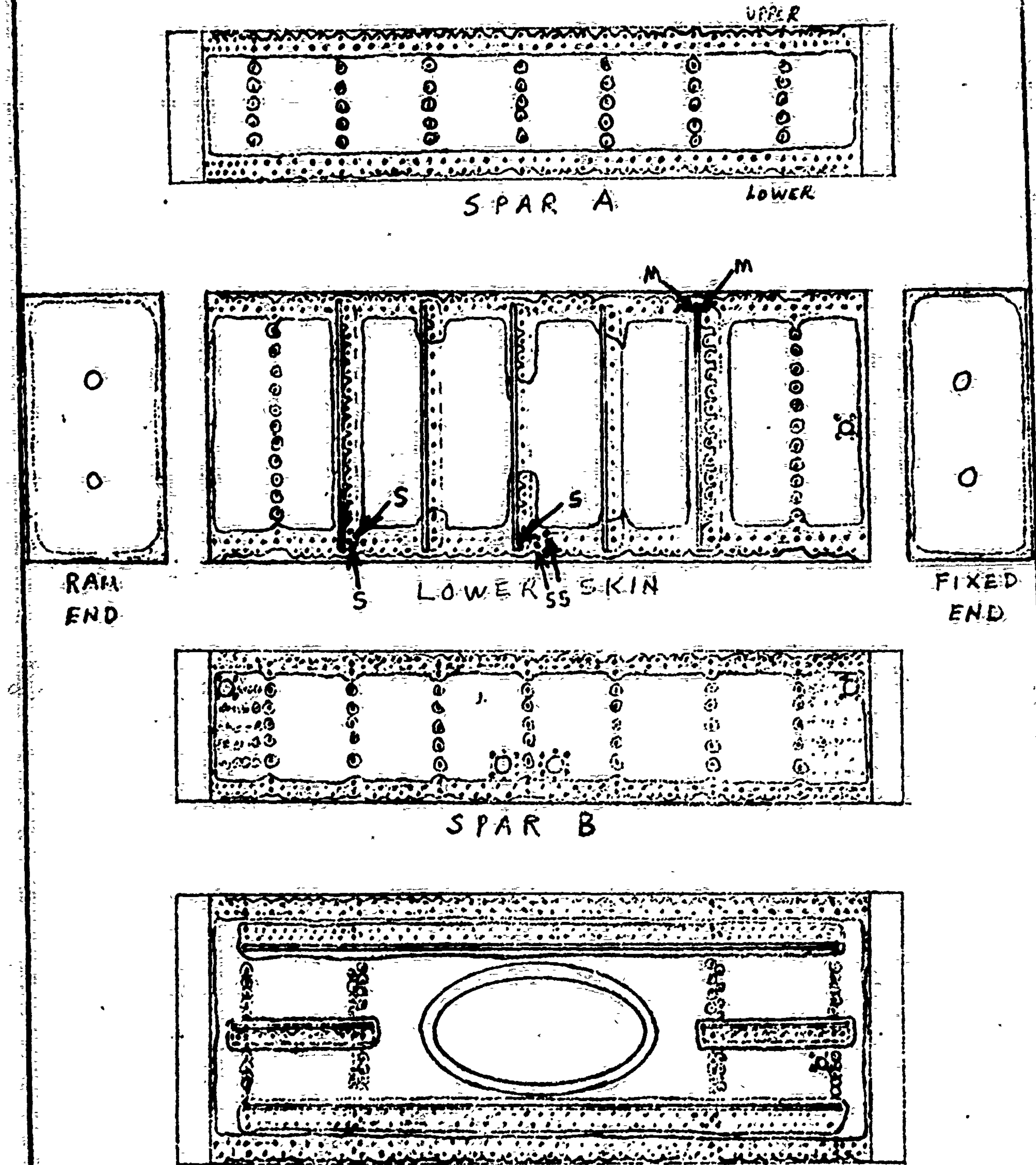
① → INDICATES FILLET CRACK (LENGTH AS NOTED IN INCHES)

② → FILLET OVER FASTENER PARTIALLY TORN OFF

③ → FILLET CRACKS THAT LENGTHENED WHEN TANK WAS DRIED OUT (16 HRS 310°F) ON AUG 5 TO PREPARE FOR REPAIRS.

④ → LOSS OF ADHESION

⑤ → NEW FILLET CRACKS WHICH DEVELOPED DURING AMBIENT STORAGE (BETWEEN 6 APRIL AND 6 AUGUST 1971) OR WHEN TANK WAS DRIED OUT (16 HRS @ 310°F) ON AUG 5, 1971 TO PREPARE FOR REPAIRS.



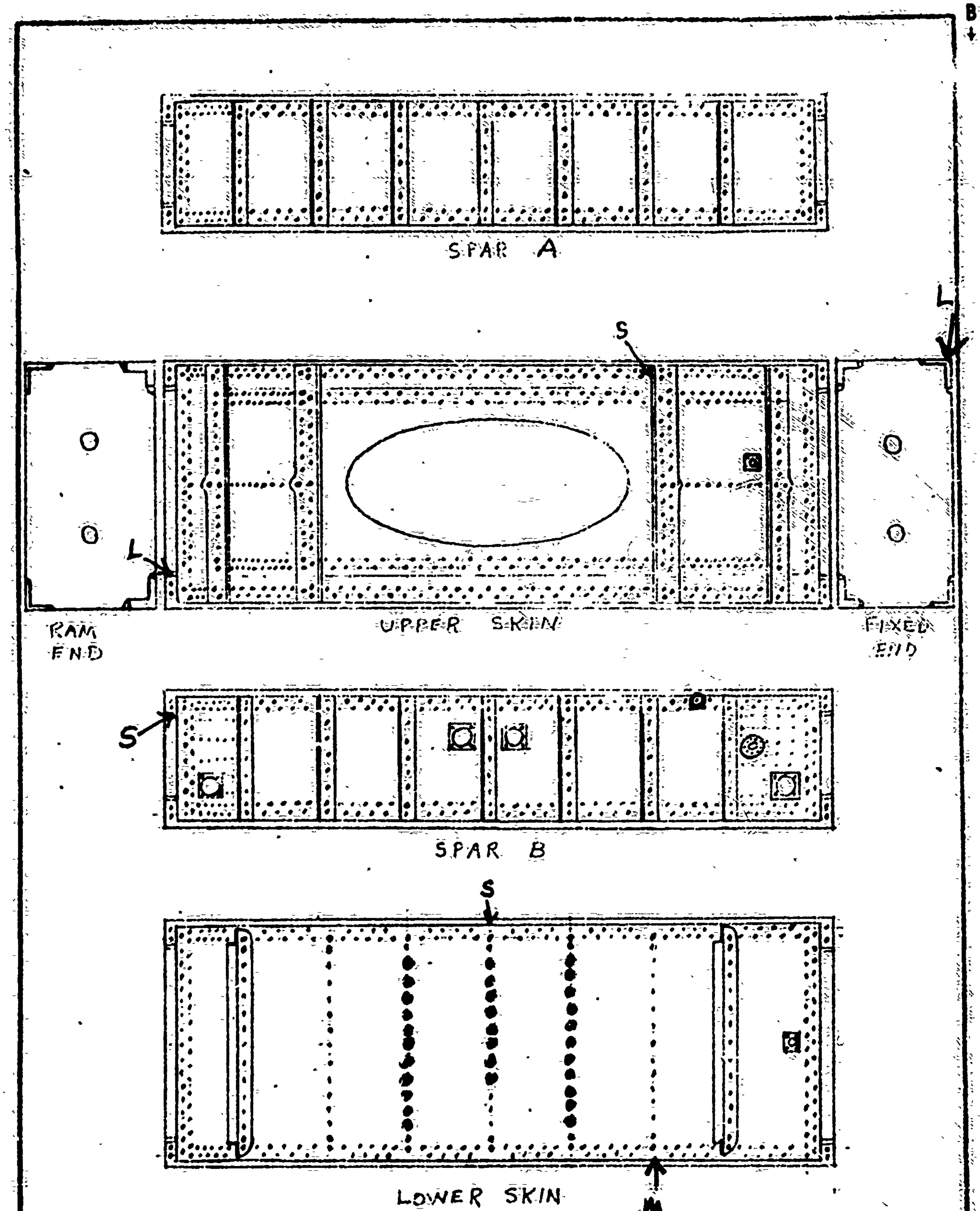
L- LARGE LEAK (RAPID BUBBLING)
M- MEDIUM LEAK (~1 BUBBLE PER 15 SEC.)
S- SMALL LEAK (<1 BUBBLE PER MIN.)
INTERIOR LEAKAGE WITH 3 PSI VACUUM

LEAKAGE ON 9-9-71

CALC			REVISED	DATE	TANK NO. 3 INTERIOR VIEW LEAKAGE AFTER 16 HR'S DRY OUT AT 310°F.	
CHECK						
APD						
APD					THE BOEING COMPANY AIRPLANE DIVISION - WICHITA BRANCH	PAGE B-9

CONTRACT NO.

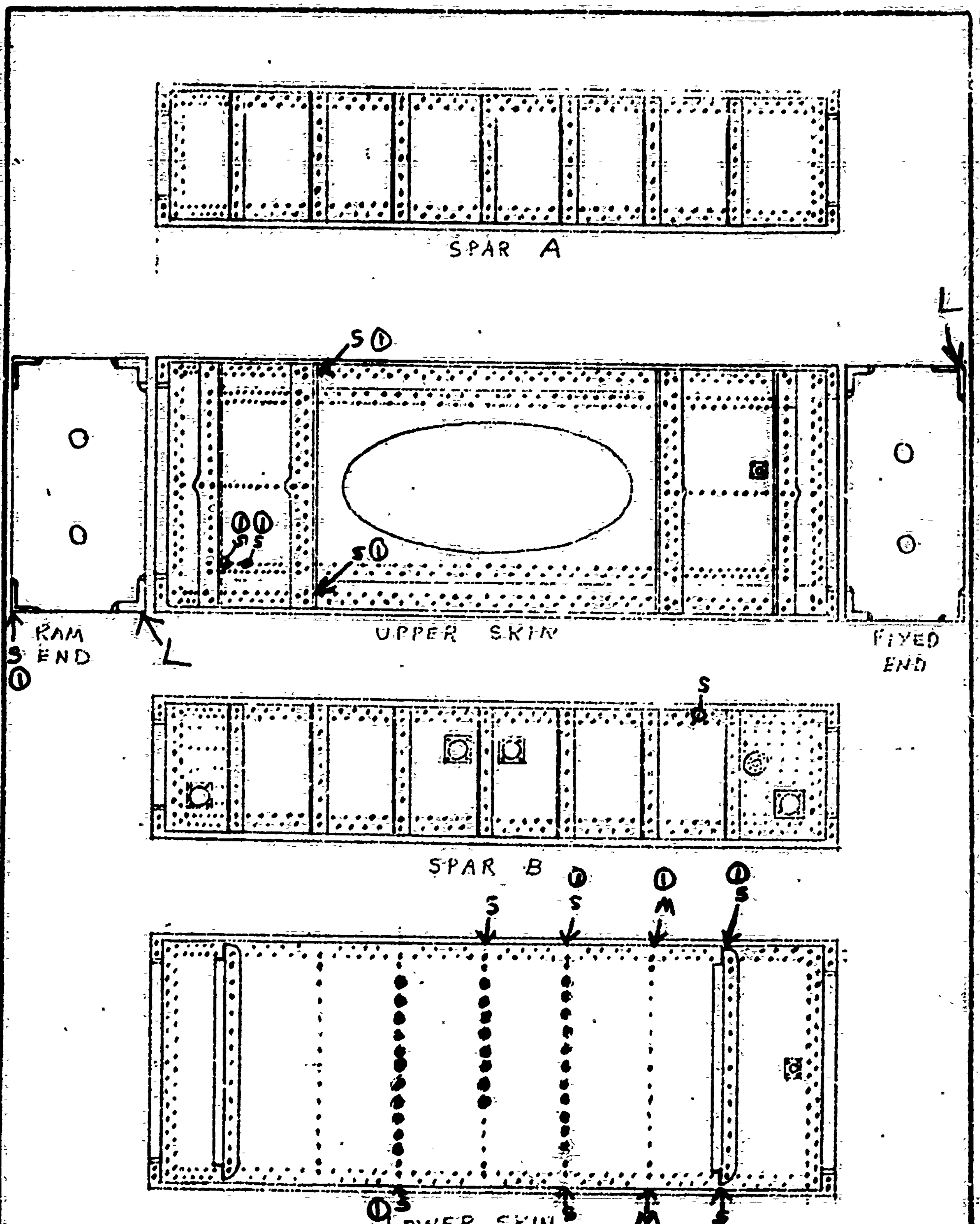
REV.



SEE PAGE B-9 FOR CODE

LEAK TESTED 8-13-71

			REVISED	DATE	
CALC					TANK NO. 3 LEAKAGE TEST NO. 6
CHECK					AFTER REPAIRING 6 LEAKING CORNERS
APR					
APR					THE BOEING COMPANY AIRPLANE DIVISION • WICHITA BRANCH
CONTRACT NO.					PAGE B-10



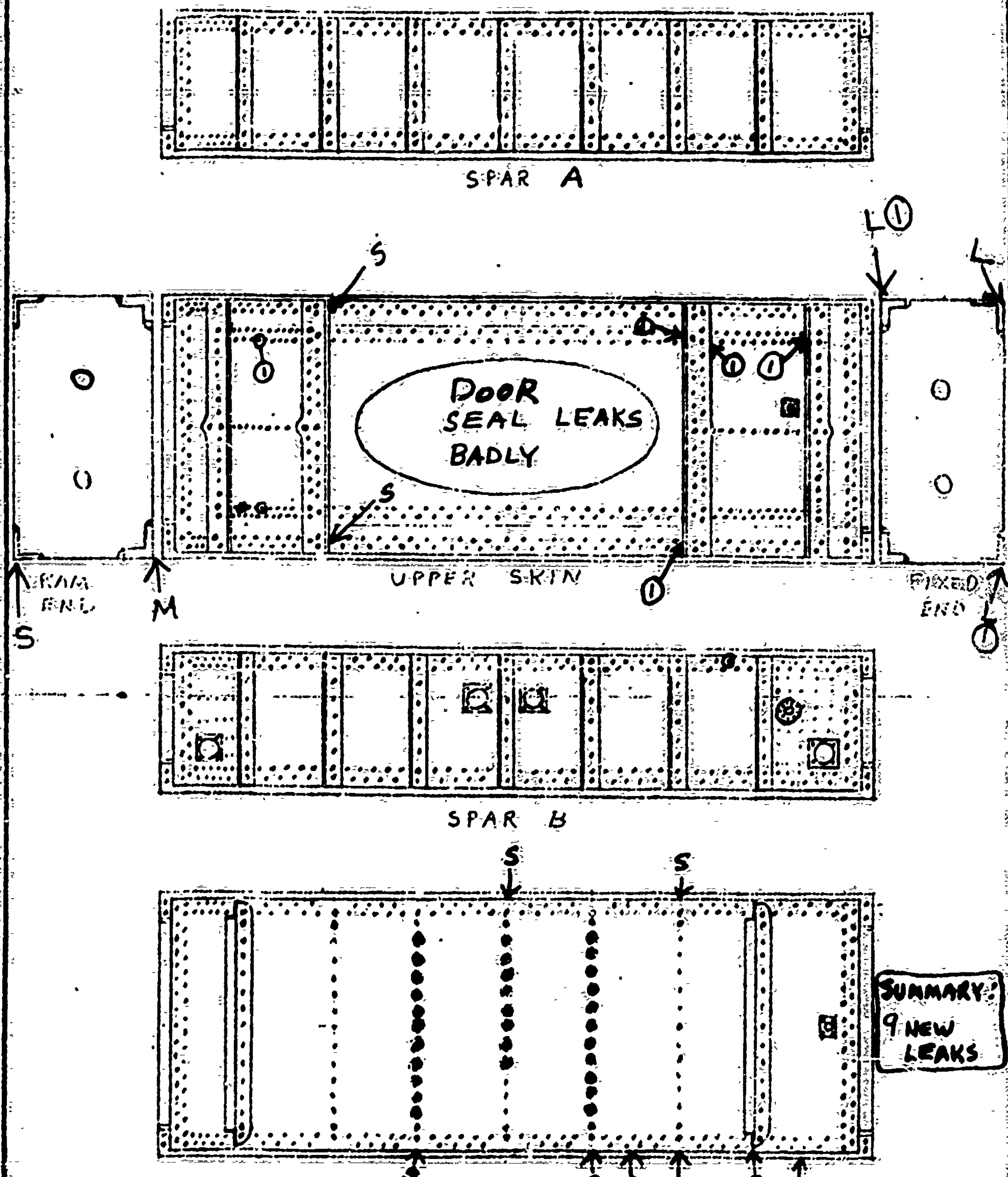
①– NEW LEAKS THIS TEST

S = SMALL LEAK
M = MED. LEAK
L = LARGE LEAK

TESTED 9-2-71

CALC	JM		REVISED	DATE	TANK NO. 3 LEAKAGE TEST NO. 6 AFTER LOAD CYCLING AT THREE TEMPERATURES (FOLLOWING 8 CYCLES
CHECK					
APR					
APR					THE BOEING COMPANY
CONTRACT NO.					OF ENVIRONMENTAL CONDITIONING)

PAGE B-11



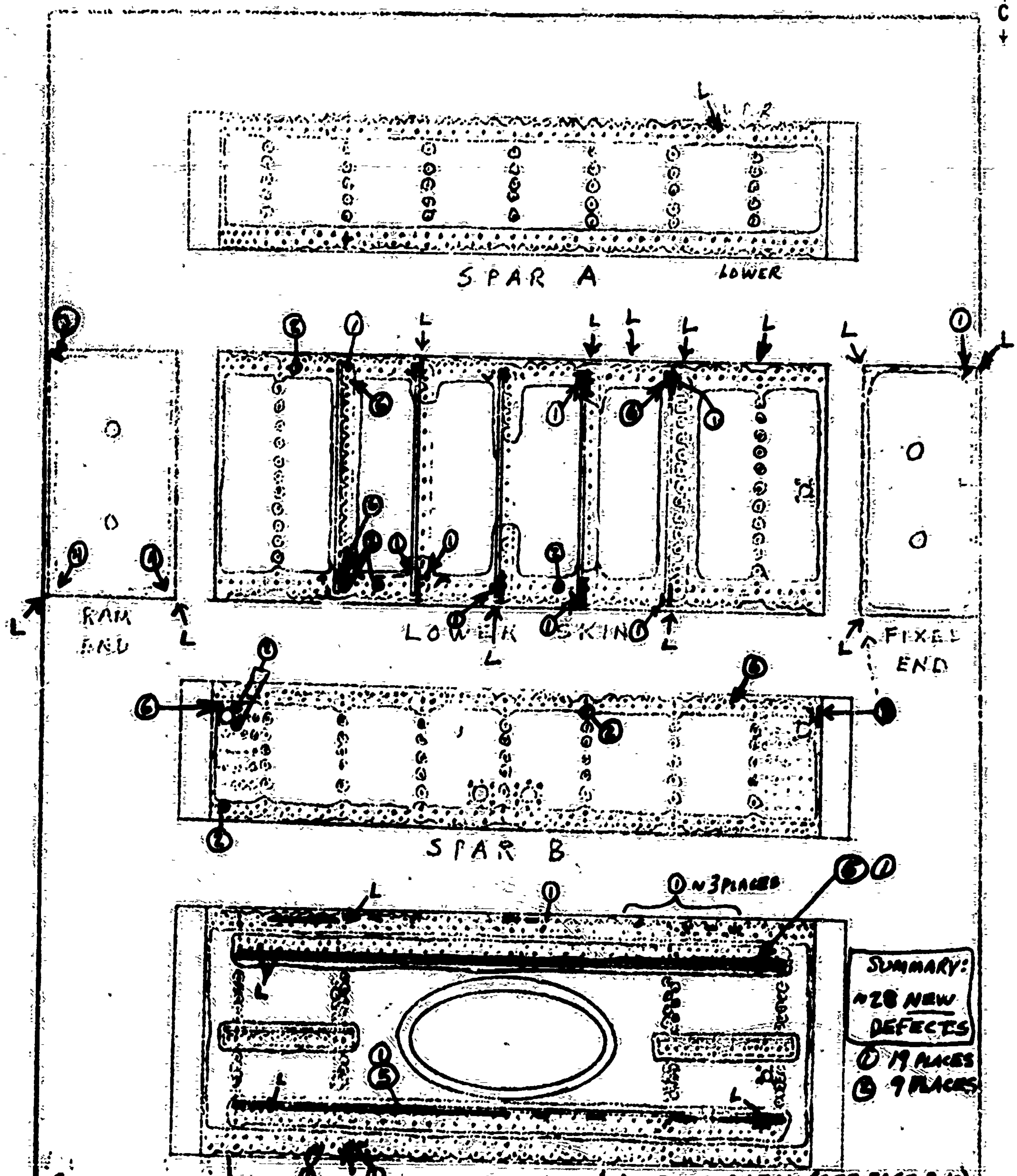
① NEW LEAKS THIS TEST

S - SMALL LEAK
M - MEDIUM LEAK
L - LARGE LEAK

TESTED 1-4-72 & 1-18-72

CALC	[illegible]	1-4-72	REVISED	DATE	TANK NO. 3 LEAKAGE TEST NO. 7
CHECK					AFTER 22 ENVIRONMENTAL
APR					CONDITIONING CYCLES
APR					THE BOEING COMPANY
					PAGE B-12

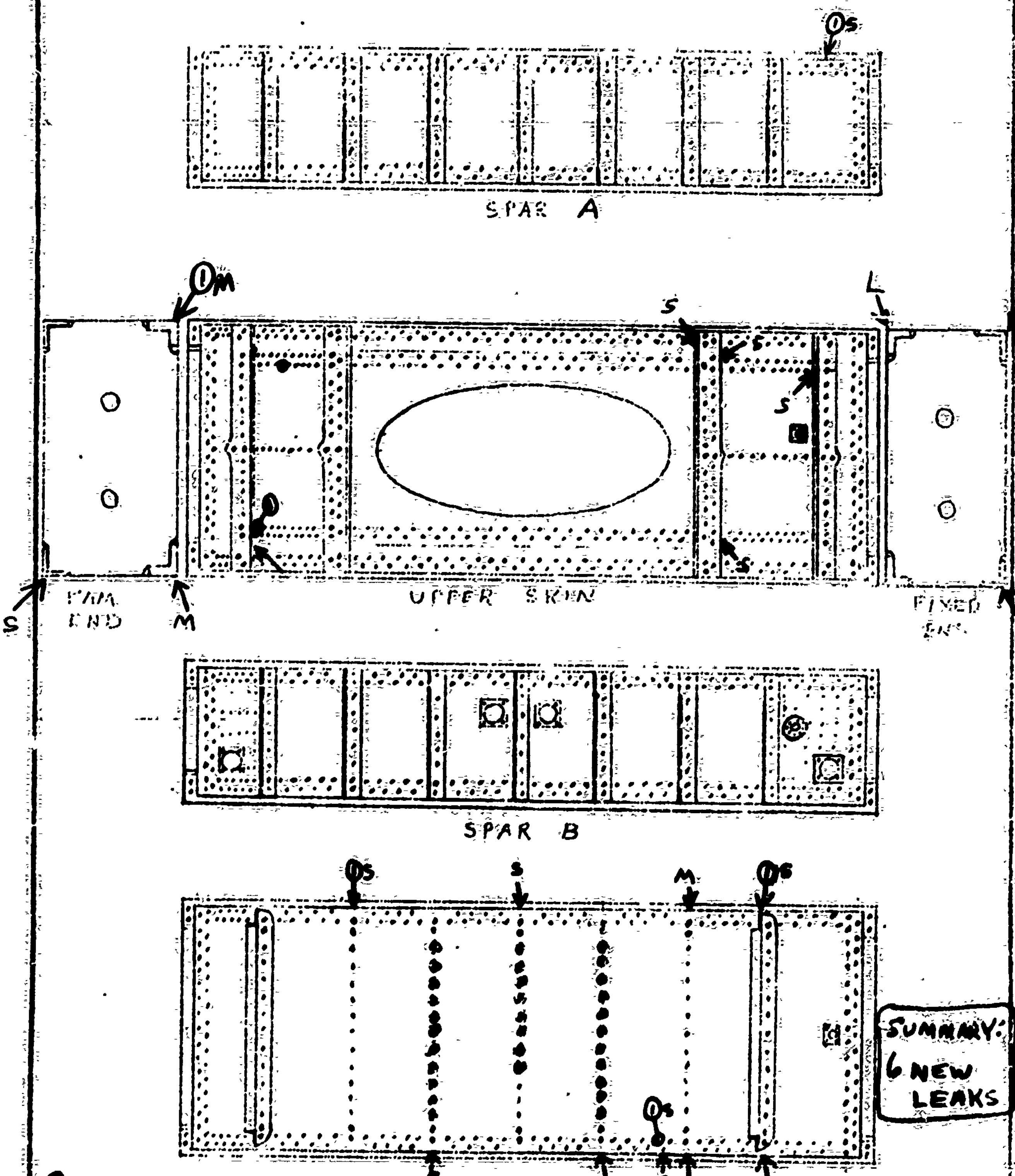
CONTRACT NO.



① NEW CRACK THIS TEST
② SEALANT OVER FASTENER TORN
③ NEW CRACK IN ORIGINAL SEALANT
L = EXTERIOR LEAK (SEE PAGE B-12)
④ SOME EVIDENCE OF LOSS OF ADHESION OF REPAIR SEALANT TO ORIGINAL SEALANT
⑤ FILLET CRACK ALMOST ENTIRE LENGTH ALONG STIFFENER

CALC			REVISED	DATE	TANK NO. 3 INTERIOR VIEW AFTER 22 ENVIRONMENTAL CONDITIONING CYCLES (JAN 72)
CHECK					
APR					
APR					THE BOEING COMPANY
CONTRACT NO.					⑥ INTERIOR LEAK AT 3 PSIG VACUUM — PAGE: B-13

REV. C



1 NEW LEAKS THIS TEST

S = SMALL LEAK

M = MEDIUM LEAK   L = LARGE LEAK

TESTED 1-26-72.

CALC			REVISED	DATE
CHECK				
APR				
APR				

TANK NO. 3 LEAKAGE TEST NO. 5
AFTER LOAD CYCLING AT 3 TEMPS.
(FOLLOWING 22 CYCLES OF ENV. COND.)

THE BOEING COMPANY

CONTRACT NO.

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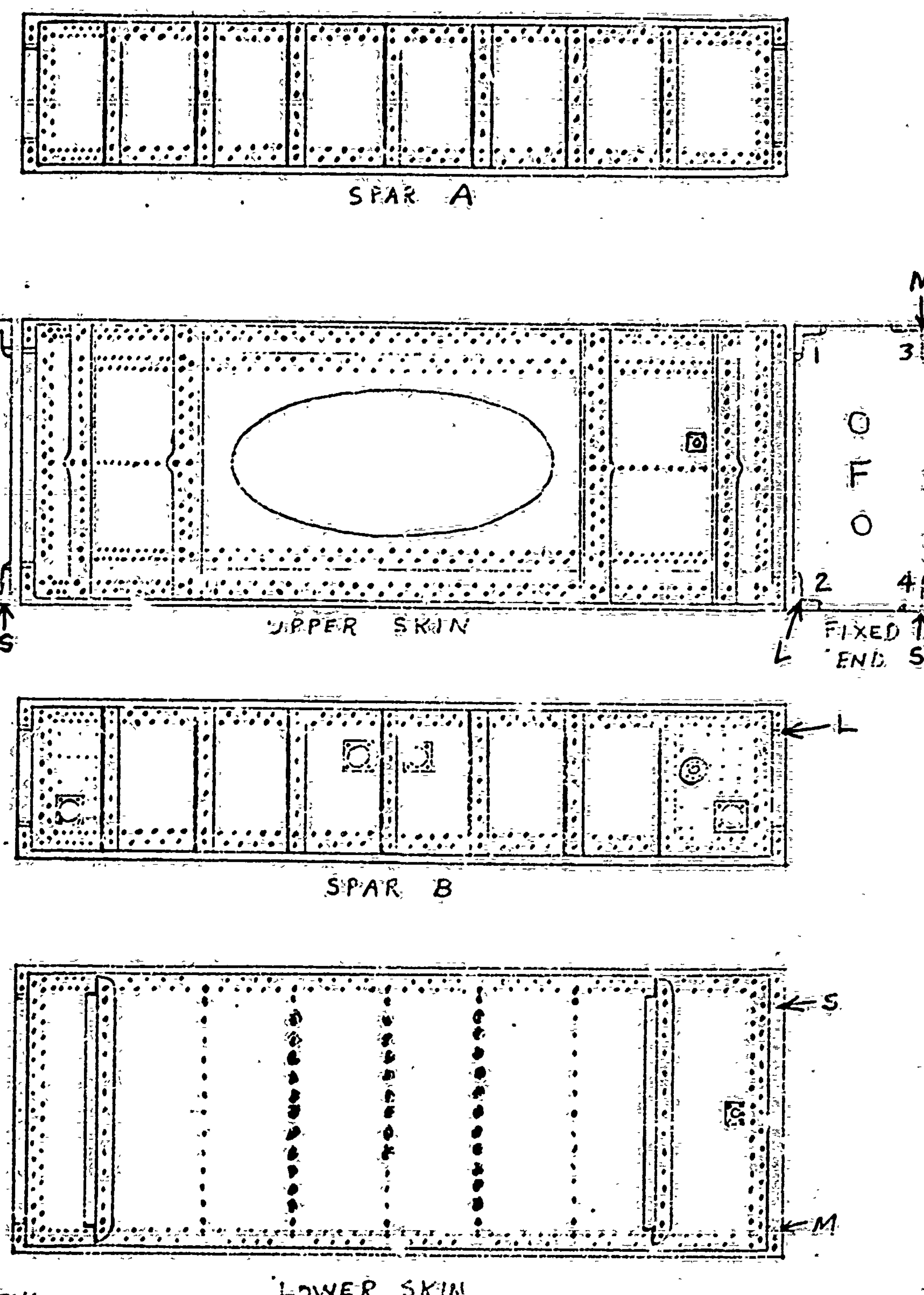
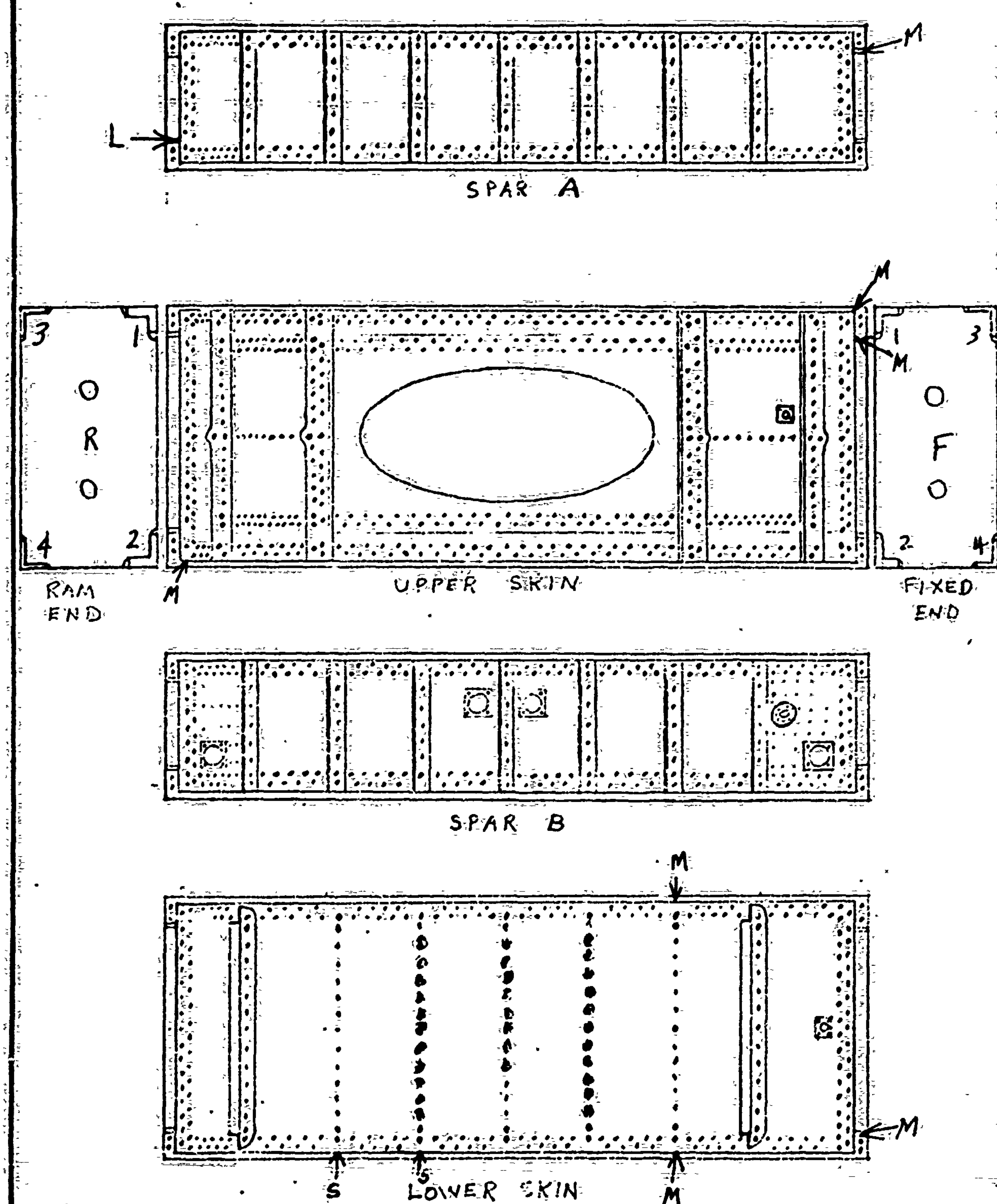


S = SMALL LEAK
M = MEDIUM LEAK
L = LARGE LEAK

TESTED 2-8-72

			REVISED	DATE	TANK NO. 3 LEAKAGE TEST NO. 1 AFTER REPAIRS OF SIX LEAKING CORNERS (SEE PAGE B-14)	
CALC						
CHECK						D3-8297
APR					THE BOEING COMPANY	
APR						PAGE B 15
CONTRACT NO.						104.10



			REVISED	DATE	
CALC					TANK NO. 3 LEAKAGE TEST NO. 10
CHECK					AFTER 33 CYCLES OF ENVIRONMENTAL
APR					CONDITIONING (SEE TABLE 7)
APR					THE BOEING COMPANY AIRPLANE DIVISION • WICHITA BRANCH

D3-8297

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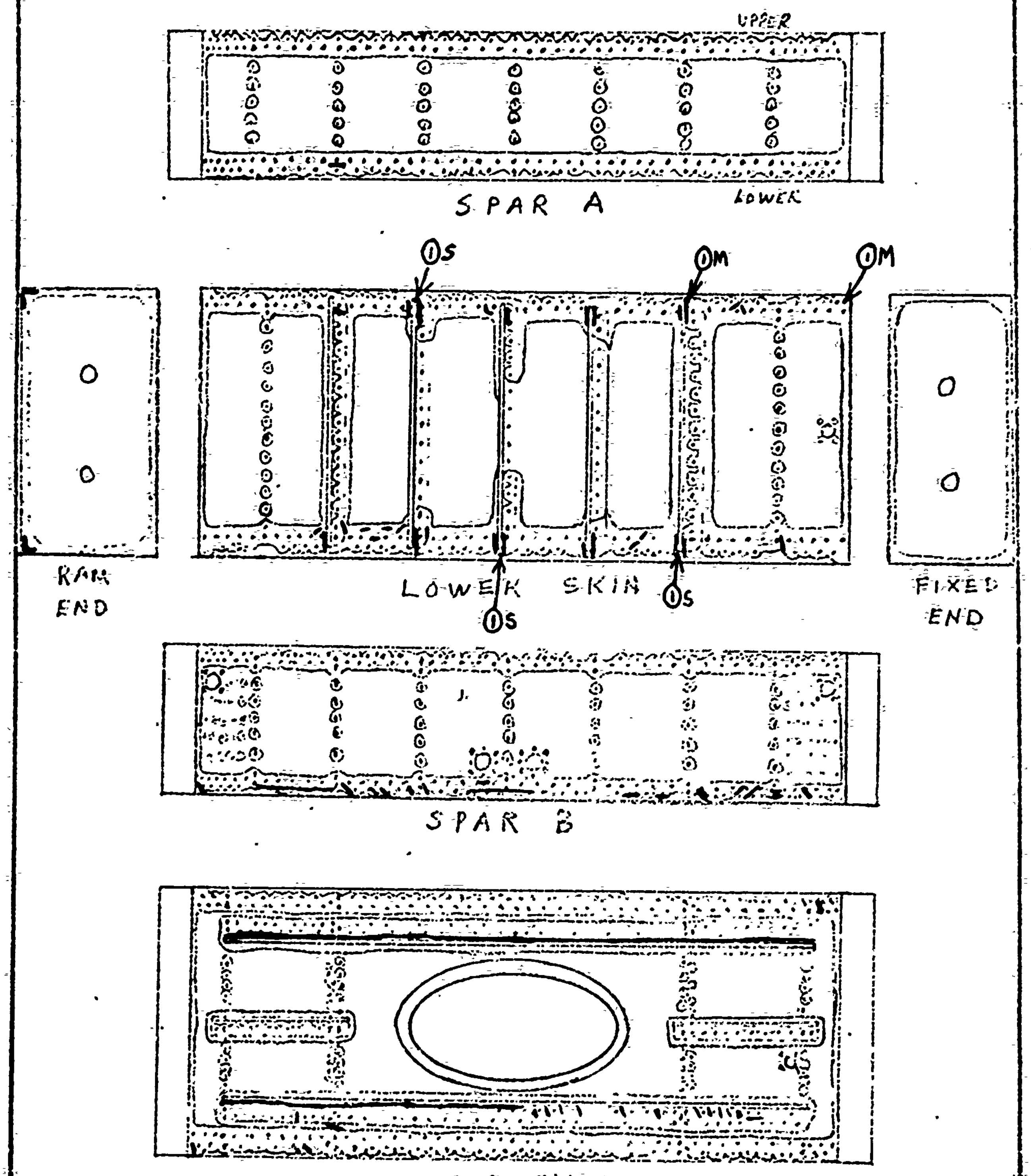
104.11

CONTRACT NO.

E-80. 14 REV. D



S = SMALL LEAK
M = MEDIUM LEAK
L = LARGE LEAK

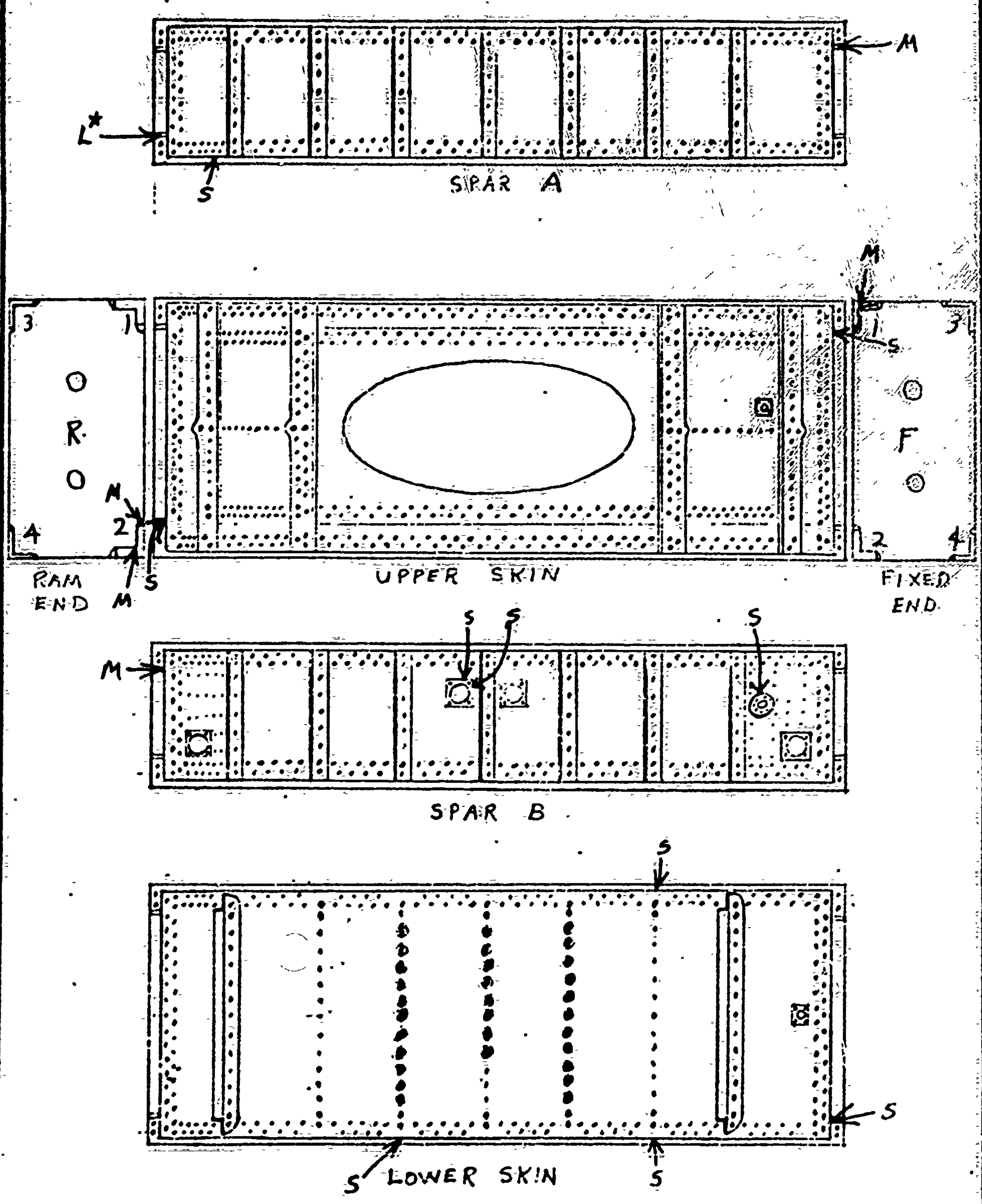
① → INTERIOR LEAK - WATER BUBBLING THRU 3 PSI VACUUM

TESTED 8-9-72

			REVISED	DATE	
CALC					TANK NO. 2 INTERIOR VIEW AFTER 39 CYCLES OF ENVIRONMENTAL CONDITIONING, PLUS 16 HOURS DRYOUT AT 400°F.
CHECK					
APR					D3-8297
APR					THE BOEING COMPANY
					PAGE B-17 104.12

CONTRACT NO.

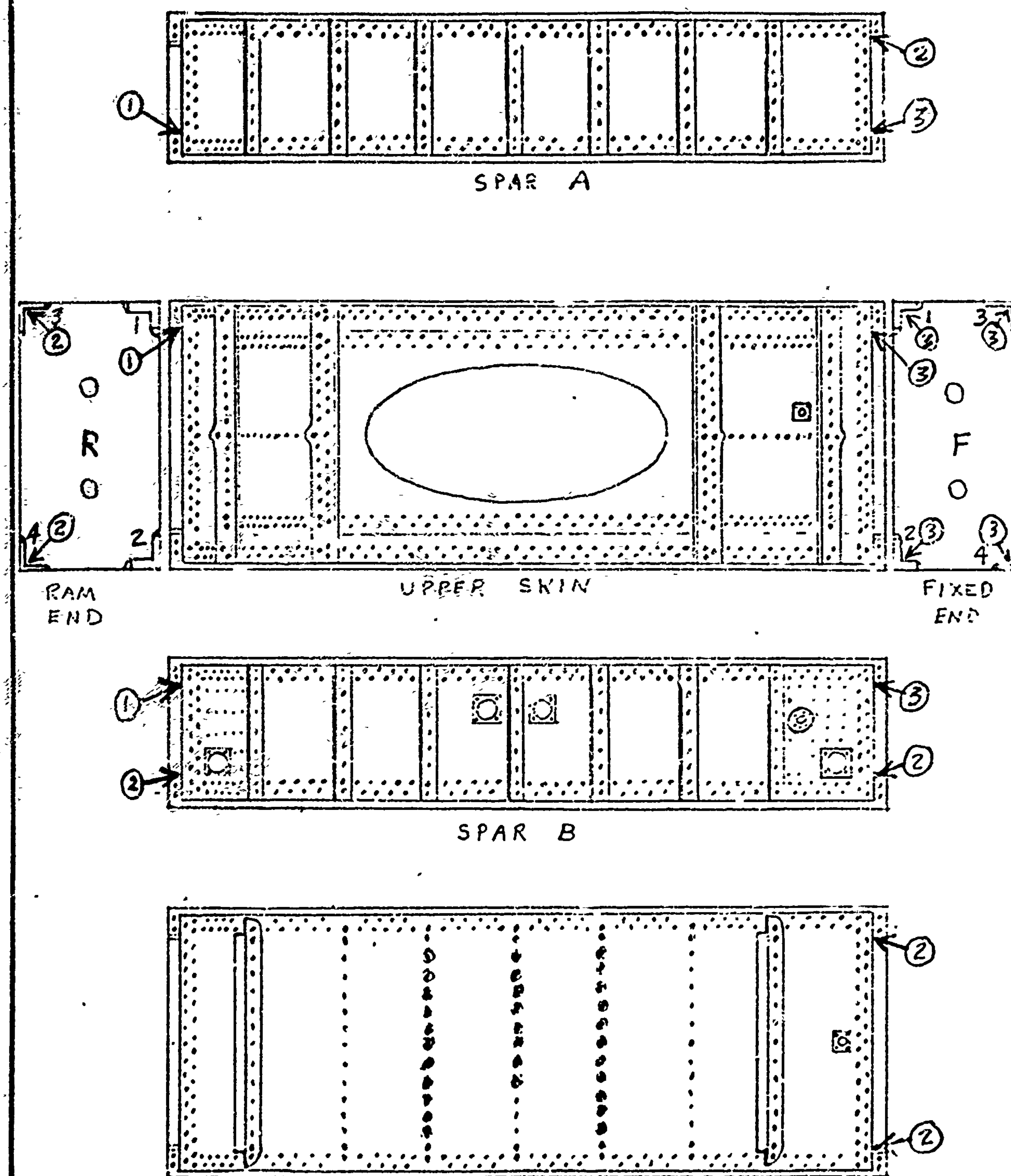
REV. D



* LEAKED STEADY STREAM WHEN FILLED WITH JP-4 ON TEST JIG | TESTED 8-16-72

			REVISED	DATE	TANK NO. 3 LEAKAGE TEST NO. 11 AFTER 39 ENVIRONMENTAL CONDITIONING CYCLES, PLUS LOAD CYCLING AT ALL 3 TEMPERATURES	
CALC						
CHECK						
APR						
APR					THE BOEING COMPANY	D3-8297
						PAGE B-18
CONTRACT NO.						104.13

E-302 R4 REV. D



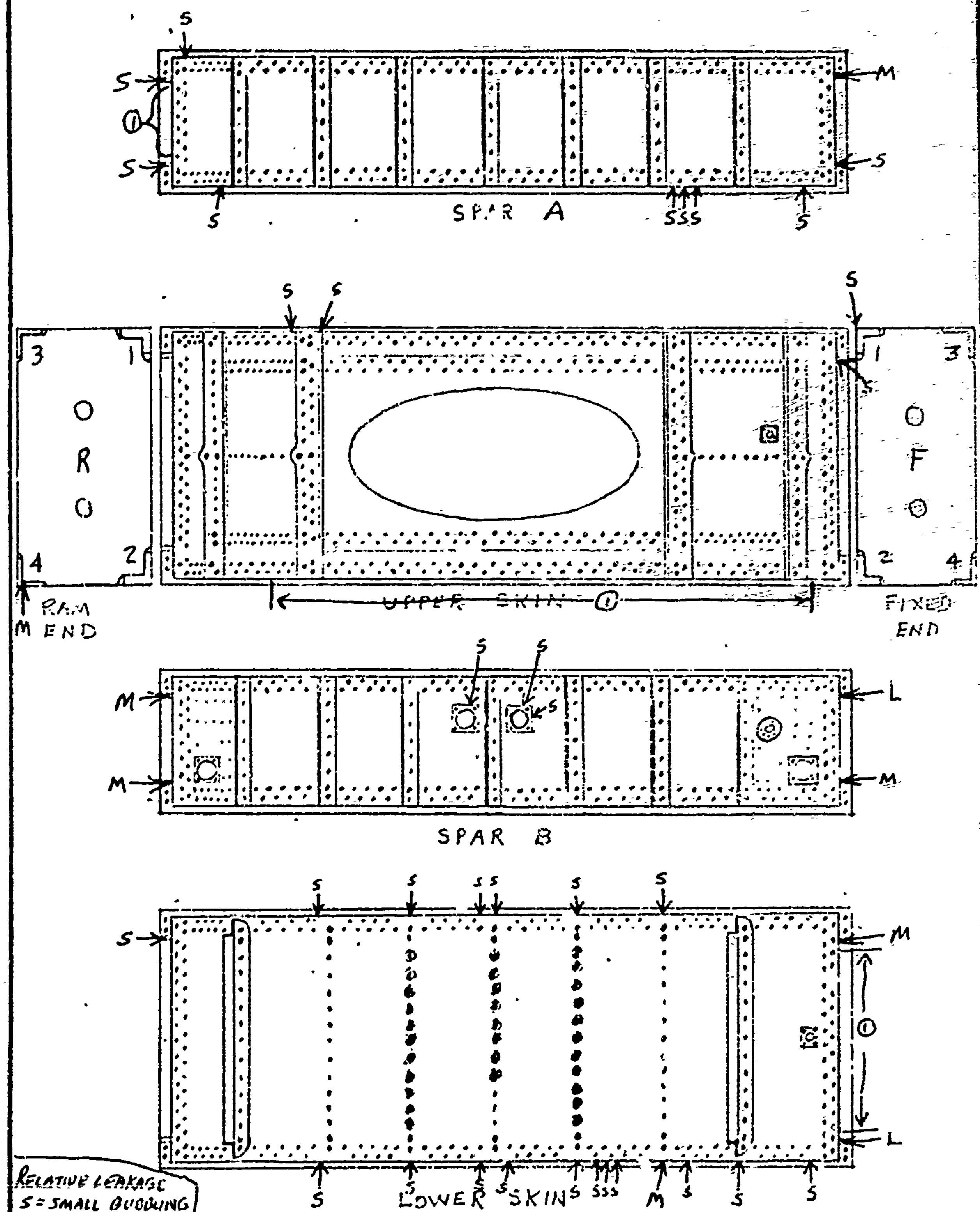
① LARGE LEAK
② SMALL LEAK
③ VERY SMALL LEAK

ENDS ONLY LEAK TESTED BY WATER IMMERSION - 5 PSI AIR PRESSURE ON [illegible]

8-11-77

			REVISED	DATE	TANK NO. 3 LEAKAGE TEST NO. 17 AFTER 46 ENVIRONMENTAL CONDITIONING CYCLES	
CALC						
CHECK						D3-8297
APR					THE BOEING COMPANY	
APR					AIRPLANE DIVISION	PAGE 104.14
CONTRACT NO.						

E-582 R4 REV. D



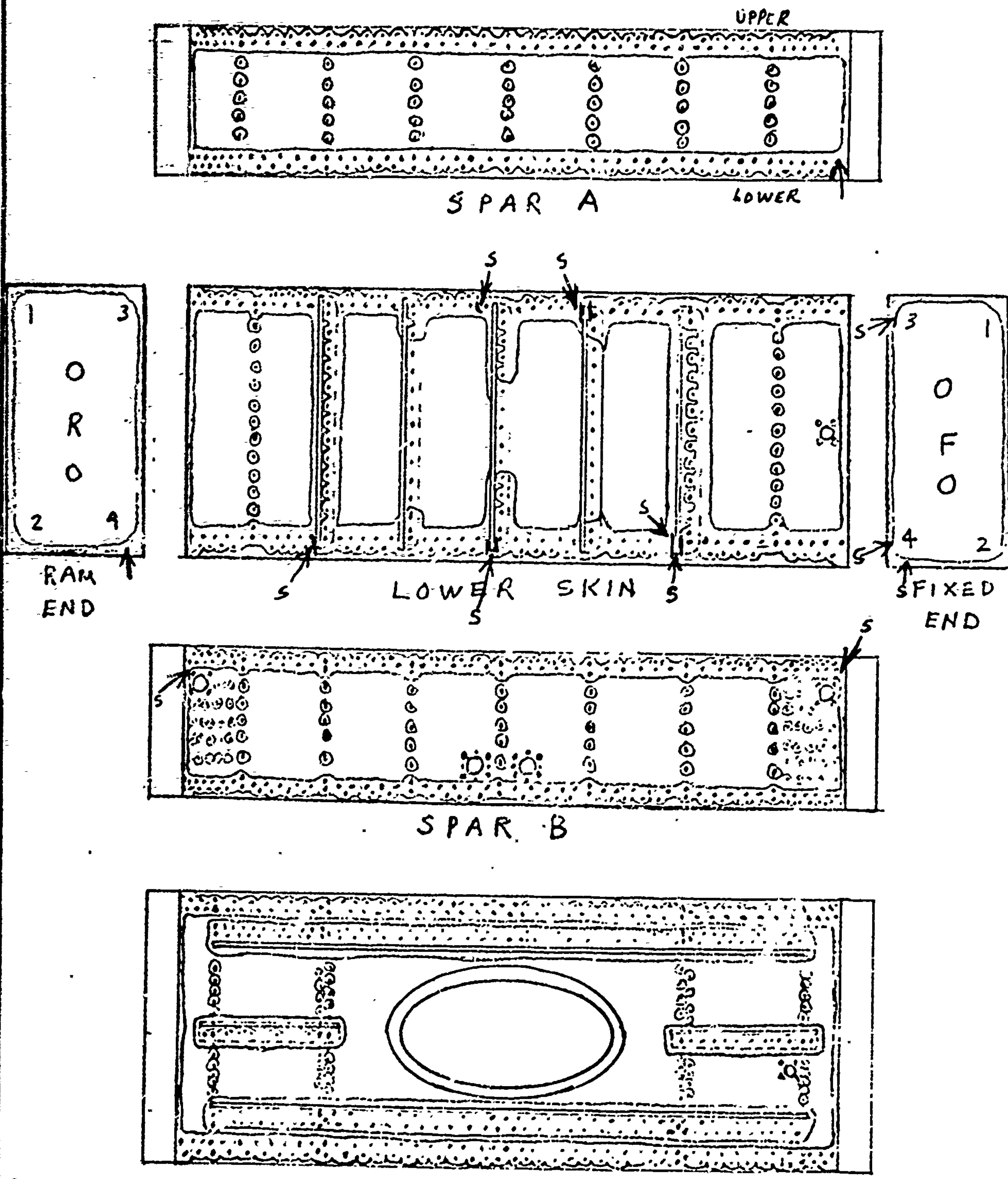
RELATIVE LEAKAGE
S = SMALL BUBBLING
M = MEDIUM "
L = LARGE "

① VERY SLIGHT BUBBLING, RANDOM OVER LENGTH INDICATED

TESTED FEB 6, 1973

			REVISED	DATE		
CALC	Lu				TANK NO. 3 LEAKAGE TEST NO. 12 AFTER 54 ENVIRONMENTAL CONDITIONING CYCLES PLUS LOADING AT ALL 3 TEMPERATURES	
CHECK						
APR						D3-8297
APR					THE BOEING COMPANY AIRPLANE DIVISION	
						PAGE B-20 104.15
CONTRACT NO.						

E-382 R4 REV. D

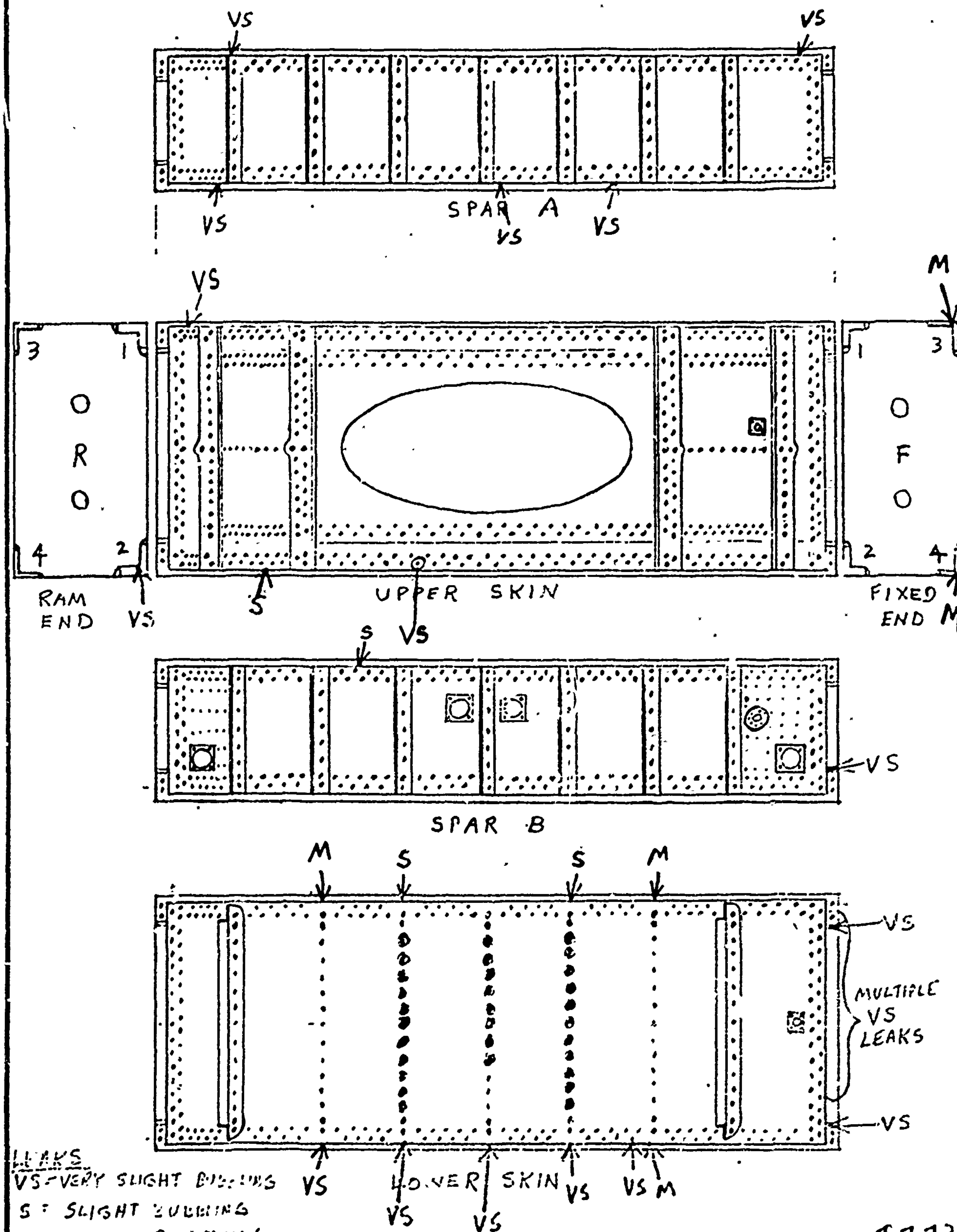


S = SMALL BUBBLING
M = MED. "
L = LARGE "

→ INTERIOR LEAKAGE - WATER BUBBLING THROUGH 3PSI VACUUM

TESTED 26-7?

CALC			REVISED	DATE	TANK NO. 3 INTERIOR LEAKAGE AFTER 54 ENVIRONMENTAL CONDITIONING CYCLES PLUS LOADING AT ALL 3 TEMPERATURES
CHECK					
APR					D3-8297
APR					THE BOEING COMPANY AIRPLANE DIVISION WICHITA BRANCH
					PAGE 5-21
CONTRACT NO. REV. D					104.16



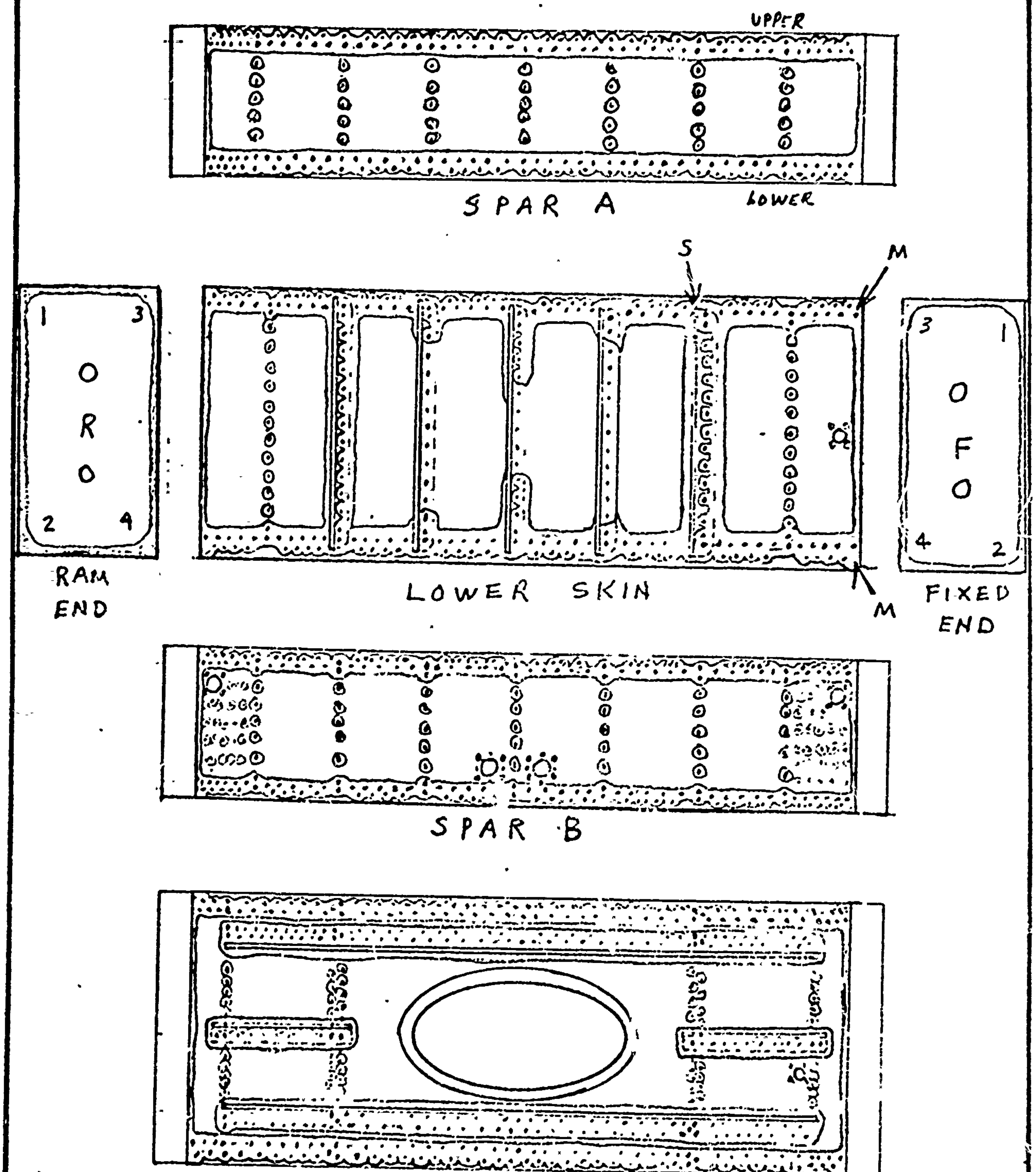
LEAKS
VS = VERY SLIGHT BUBBLING
S = SLIGHT BUBBLING
M = MEDIUM BUBBLING

8-7-73

			REVISED	DATE		
CALC					TANK NO. 3 LEAKAGE TEST NO. 1	
CHECK					A.I.R. 74 ENVIRONMENTAL CONDITIONING [illegible]	
APR					[illegible]	D3-8297
APR					THE BOEING COMPANY [illegible]	
						PAGE [illegible]
CONTRACT NO.						

E-582-R4 REV. D

104.17



LEAKS
S = SMALL BUBBLING
M = MEDIUM BUBBLING

8-7-73

CALC			REVISED	DATE	TANK NO. 3 INTERIOR VIEW LEAKS: AFTER 71 ENVIRONMENTAL CONDITIONING CYCLES [illegible]	
CHECK						
APR						
APR					THE BOEING COMPANY AEROSPACE DIVISION WICHITA BRANCH	D3-8297
						PAGE B-23
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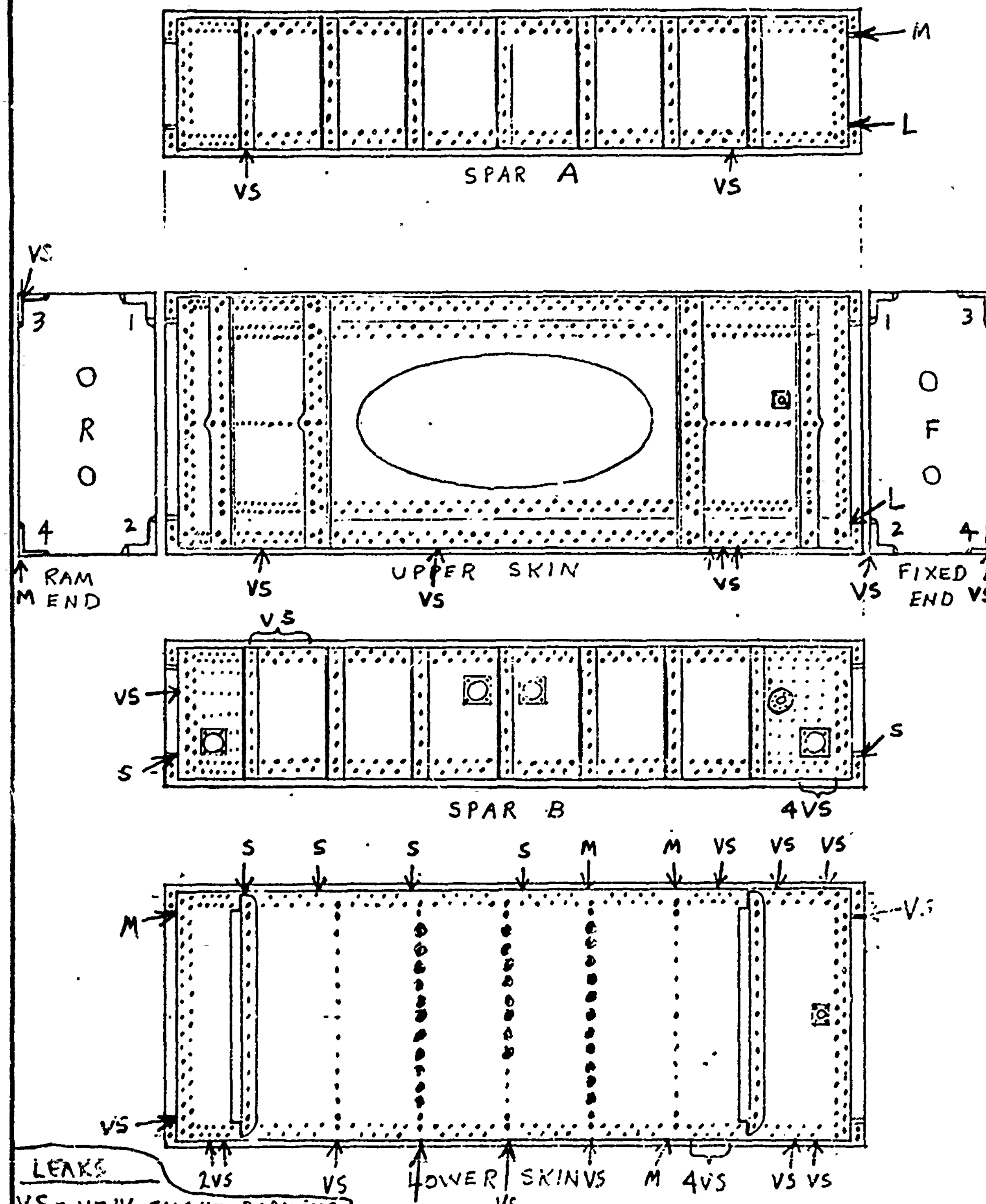
REV. B

104.18



LEAKS

VS - VERY SLIGHT BUBBLING

S - SLIGHT BUBBLING

M - MEDIUM BUBBLING L - LARGE BUBBLING

1-15-74

CALC			REVISED	DATE	TANK NO. 3 LEAKAGE TEST NO. 14 AFTER 92 ENVIRONMENTAL CONDITIONING CYCLES	
CHECK						
APR						D3-8297
APR					THE BOEING COMPANY AIRPLANE DIVISION WICHITA BRANCH	PAGE B-24
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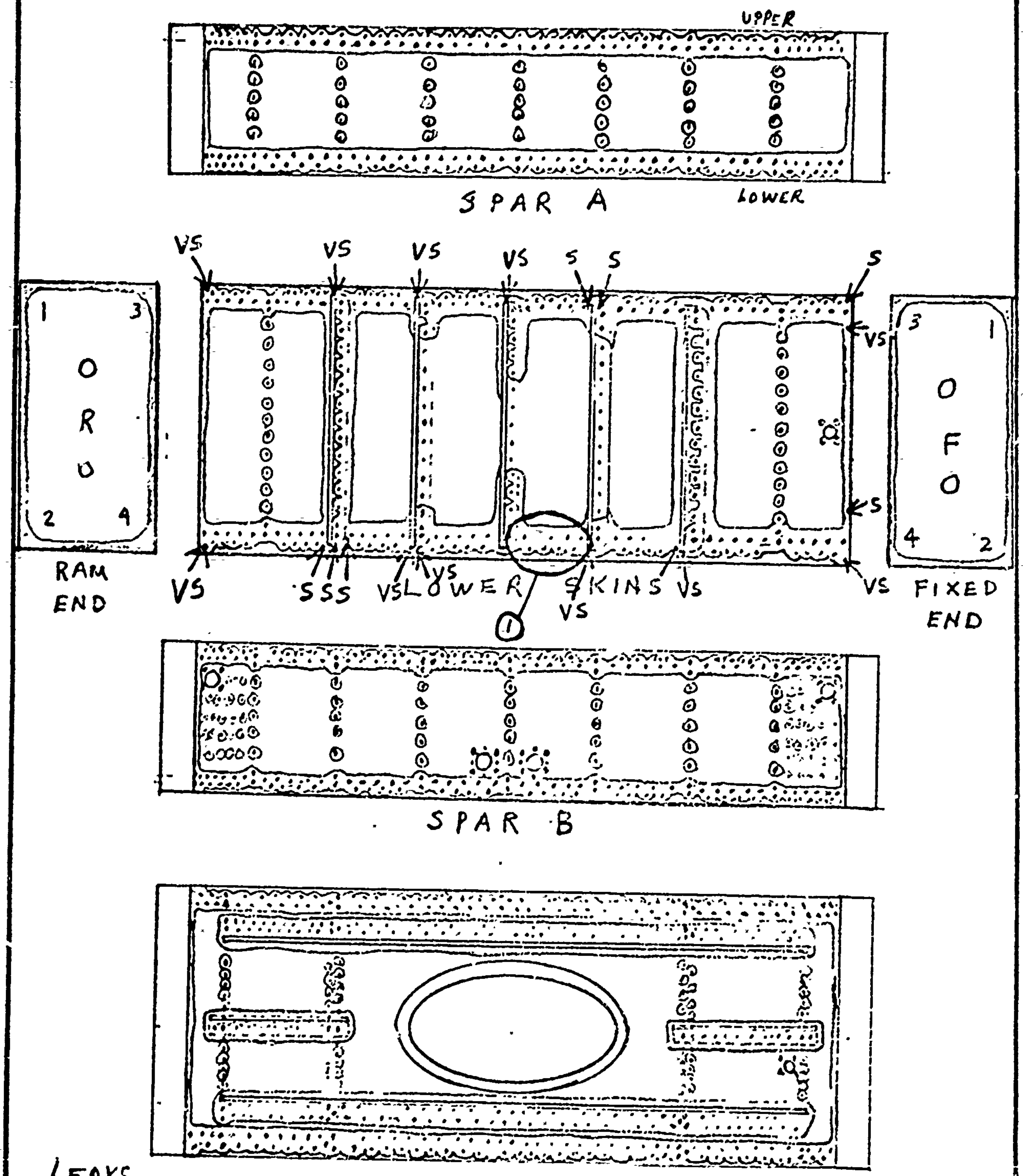

LEAKS

VS- VERY SMALL BUBBLING

S- SMALL BUBBLING

(1) AREA STRIPPED AND RESEALED ON 1-16-74.

1-15-74

			REVISED	DATE	TANK NO. [illegible] INTERIOR VIEW LEAKAGE AFTER 92 ENVIRONMENTAL CONDITIONING CYCLES	
CALC						
CHECK						
APR						D3-8297
APR					THE BOEING COMPANY AIRPLANE DIVISION WICHITA BRANCH	
						PAGE B-25
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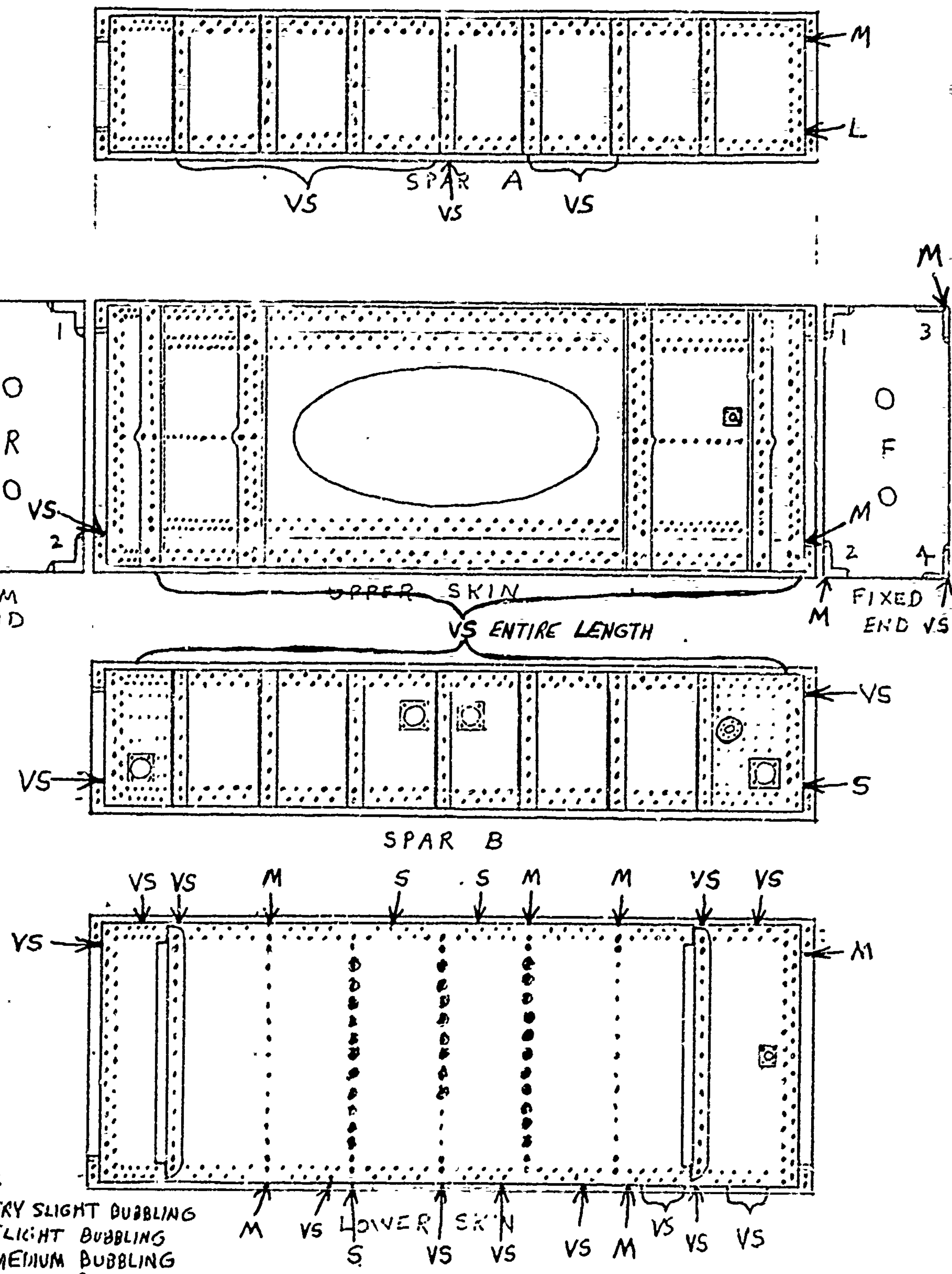
REV. B

T04.70



LEAKS
VS - VERY SLIGHT BUBBLING
S - SLIGHT BUBBLING
M - MEDIUM BUBBLING
L - LARGE BUBBLING

7-30-74

CALC	Jew	7-31-74	REVISED	DATE	TANK NO. ? LEAKAGE TEST NO. 15 AFTER 117 ENVIRONMENTAL CONDITIONING CYCLES	D3-8297
CHECK						
APR						
APR					THE BOEING COMPANY AIRPLANE DIVISION · WICHITA BRANCH	
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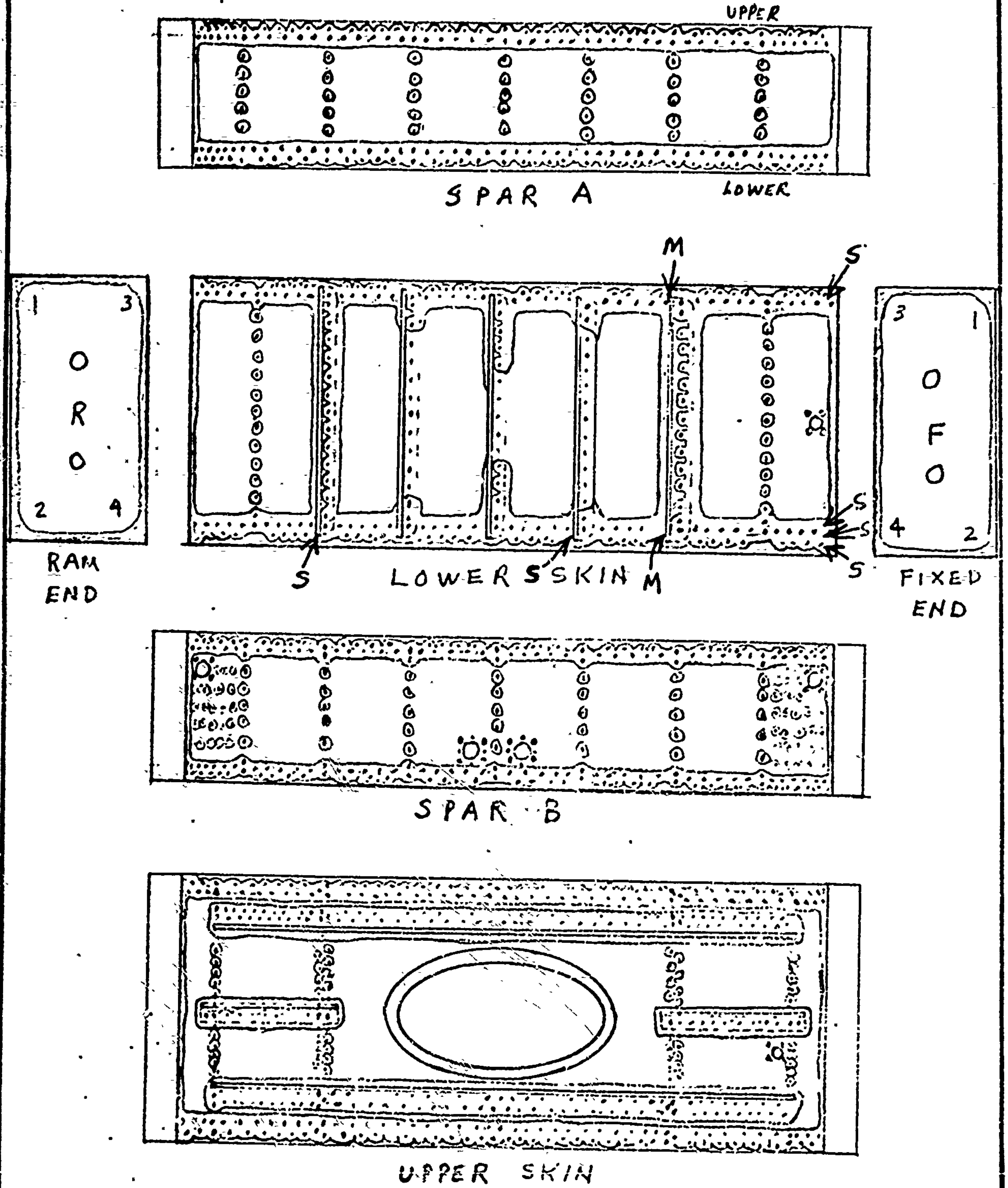

7-30-74

CALC	[illegible]	7-31-74	REVISED	DATE
CHECK				
APR				
APR				
CONTRACT NO.	Rev. E			

TANK NO. 3 INTERIOR VIEW
LEAKAGE AFTER 117 ENVIRONMENTAL
CONDITIONING CYCLES

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AIRPLANE DIVISION WICHITA BRANCH

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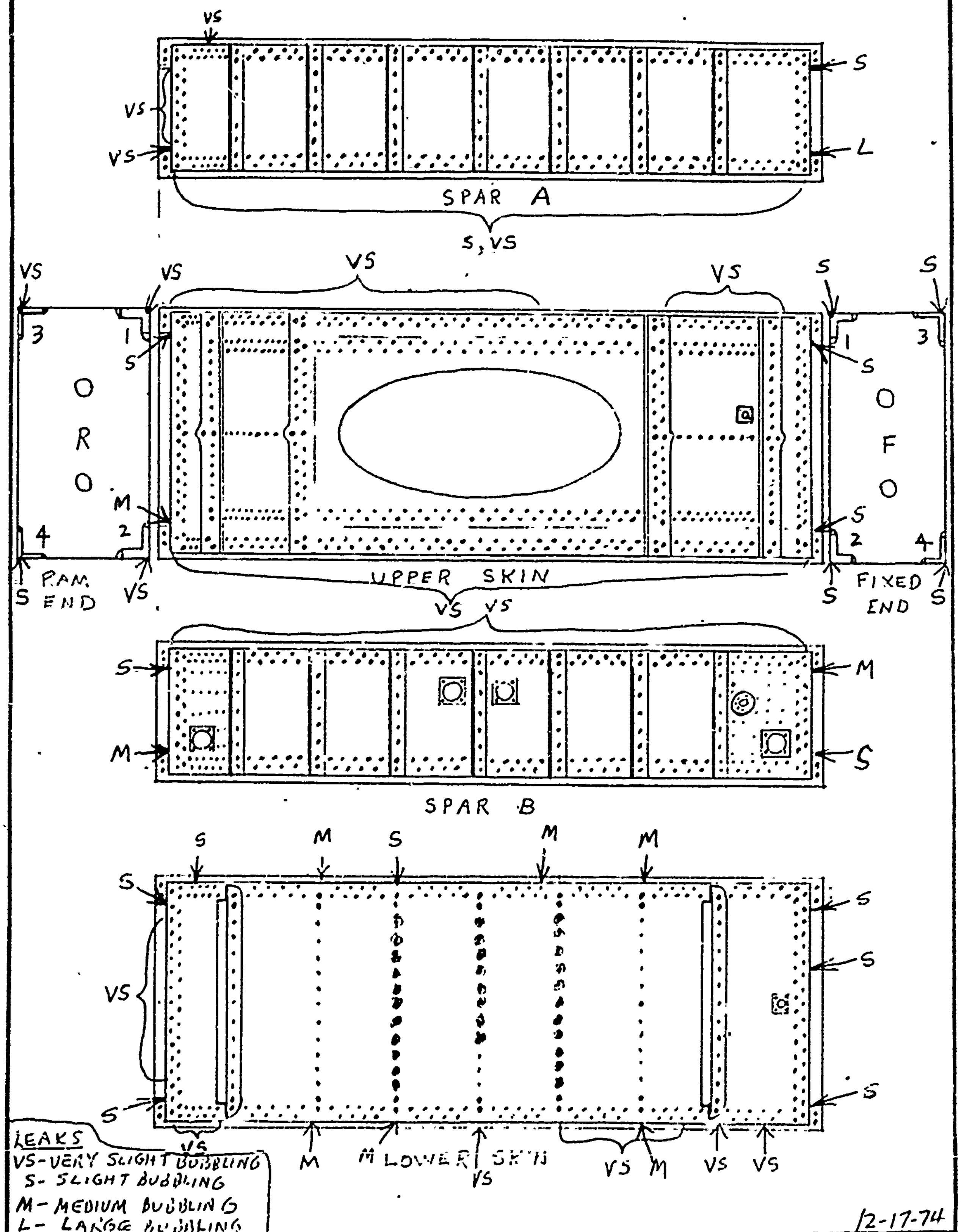
104.27

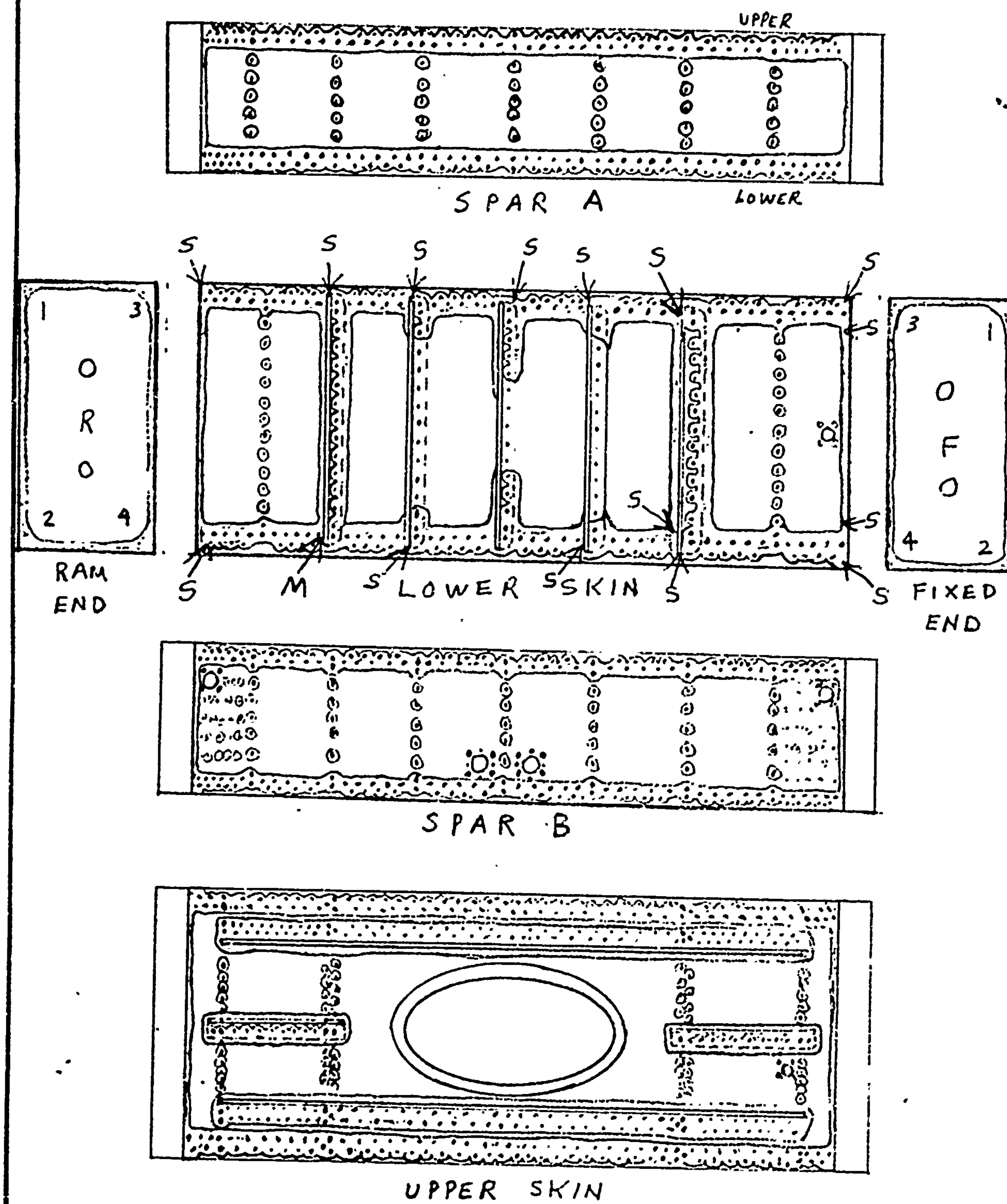


12-17-74

CALC	-m	12-17-74	REVISED	DATE	TANK NO. 3 LEAKAGE TEST NO. 16 AFTER 129 ENVIRONMENTAL CONDITIONING CYCLES	
CHECK						
APR						D3-8297
APR					THE BOEING COMPANY AIRPLANE DIVISION WICHITA BRANCH	
						PAGE B-28
CONTRACT NO.						

L-5A2 R4 Rev. E

104.23



12-17-74

CALC	[signature]	12-17-74	REVISED	DATE
CHECK				
APR				
APR				

TANK NO. 3 INTERIOR VIEW
LEAKAGE AFTER 129 ENVIRONMENTAL CONDITIONING CYCLES

D3-8297

THE BOEING COMPANY
AIRPLANE DIVISION WICHITA BRANCH

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CONTRACT NO.

A

APPENDIX C

MEASUREMENT OF WEB SHEAR WRINKLES

OF TANK NUMBER 3

MEASUREMENT OF WEB SHEAR WRINKLES OF TANK NO. 3

Reference: Coordination Sheet No. SS1-Wing-661, Schuler to Beckmann, dated 4 November 1970, Subject: "Pad-Ups of Fuel Tank Shear Webs.

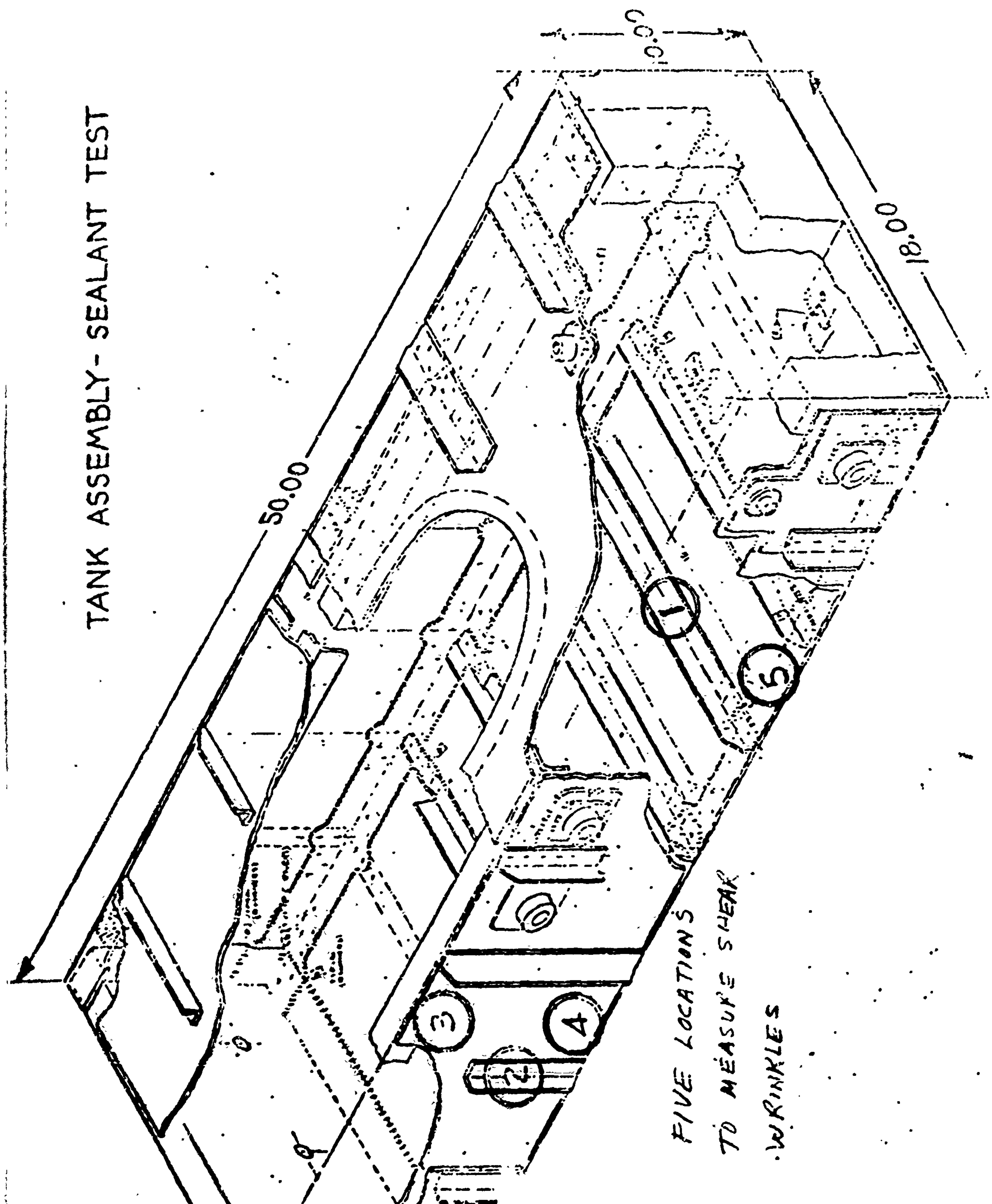
Task A. Determine torque to obtain initial web buckling (estimated to be 52,000 inch-pounds).

1. At 40,000 inch-pounds - no wrinkling.
2. At 50,000 inch-pounds - very slight buckling.
3. At 60,000 inch-pounds - definite buckling.

Task B. Determine extent and depth of web shear wrinkles at the five areas specified in the above reference. Measure at 100,000 and 216,000 inch-pounds torque in both directions.

1. This data is recorded in the attached 5 data sheets. Collecting and recording the desired information was difficult and, as can be seen, is confusing. It is suggested that Lyle Middleton (telephone 8-435-2337) be contacted if necessary for clarification.
2. In general, the results show that at 216,000 inch-pounds torque, significant web wrinkles do extend into the pad-up areas and even into the chords and stiffeners.

TANK ASSEMBLY - SEALANT TEST



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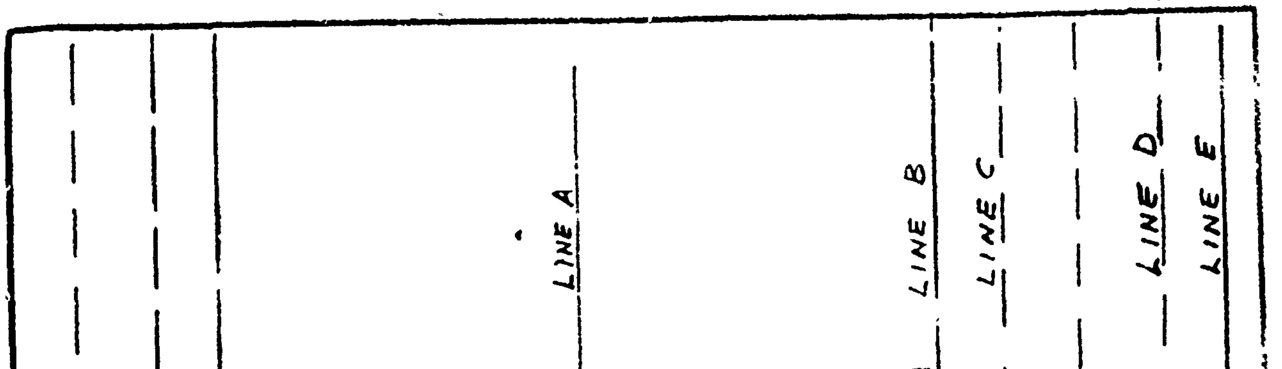


DEPTH OF WRINKLES ALONG LINES A THROUGH E WERE MEASURED WITH A $7\frac{1}{4}$ INCH LONG STRAIGHT EDGE AT THE FOLLOWING LOADS:

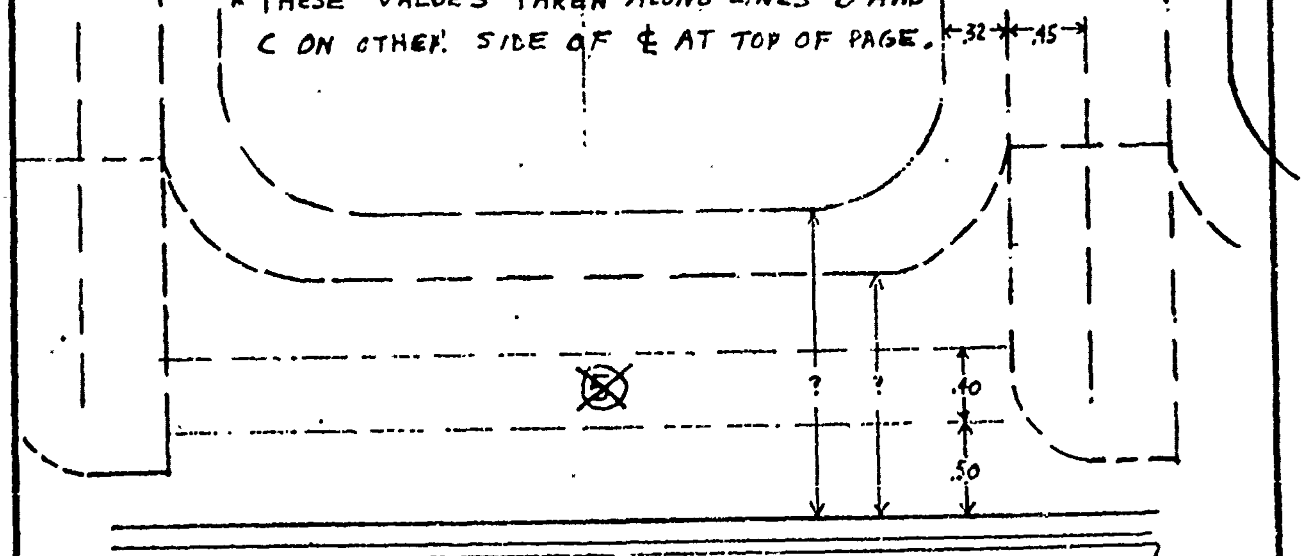
TORQUE LOAD, IN-LB:	WRINKLE DEPTH IN MILS AT LINE A	B	C	D	E	JOINT DEFLECTIONS AT LINE C	D
0	<3	<3	<3	<3	<3	<3	<3
-100,000	<3	<3	<3	<3	<3	<3	<3
-216,000	L	28	14	<3	7	<3	<3
+100,000	~5	<3	<3	<3	<3	<3	<3
+216,000	L	28*	12*	3	16	<3	<3

– IS COUNTERCLOCKWISE TORQUE
+ IS CLOCKWISE TORQUE
L IS LARGE   < IS LESS THAN

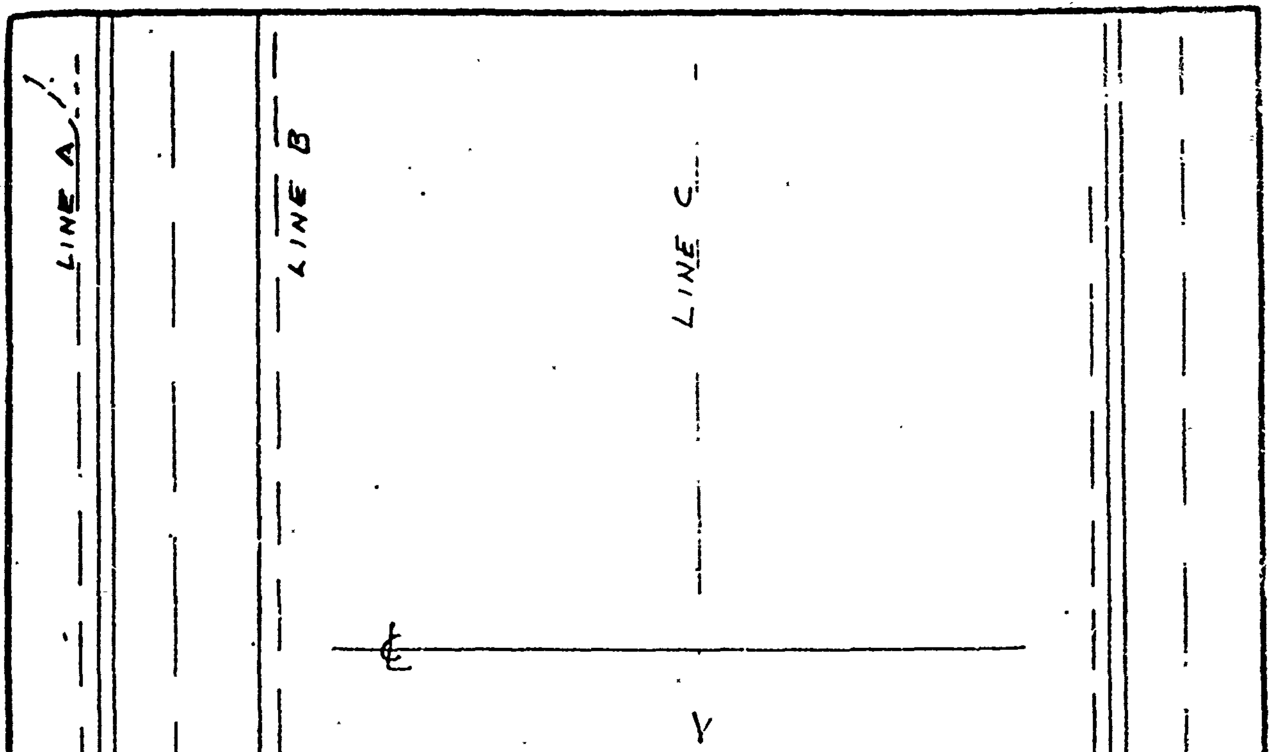
WRINKLE MEASUREMENTS TAKEN FROM TANK EXTERIOR

JOINT DEFLECTIONS TAKEN FROM TANK INTERIOR

* THESE VALUES TAKEN ALONG LINES B AND C ON OTHER SIDE OF ℄ AT TOP OF PAGE.



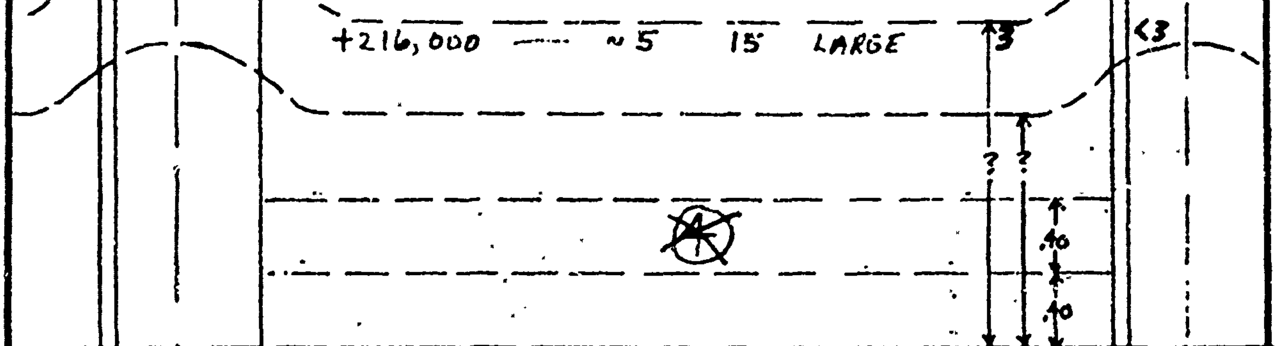
CALC			REVISED	DATE	LOCATION ① (VIEWED FROM TANK EXTERIOR)
CHECK					
APR					THE BOEING COMPANY AIRPLANE DIVISION · WICHITA BRANCH
APR					PAGE C-3
CONTRACT NO.					



②

DEPTH OF WRINKLES ON TANK EXTERIOR ALONG LINES A, B, AND C WERE MEASURED WITH A $7\frac{1}{4}$ INCH LONG STRAIGHT EDGE AT THE FOLLOWING LOADS:

TORQUE LOAD IN-LBS	WRINKLE DEPTH AT LINES A	B	C	JOINT DEFLECTION AT STIFFENER LEFT EDGE	RIGHT EDGE
0	<3	<3	3	<3	<3
-100,000	<3	<3	<3	<3	<3
-216,000	8	8	LARGE	3	<3
+100,000	<3	<3		<3	<3
+216,000	~5	15	LARGE	3	<3



CALC			REVISED	DATE	LOCATION ②
CHECK					
APR					
APR					THE BOEING COMPANY
CONTRACT NO.					

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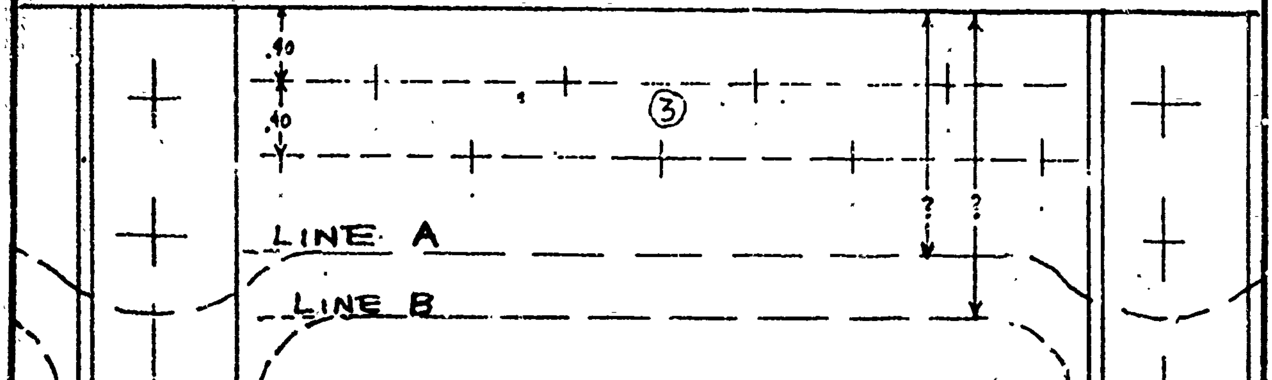


DEPTH OF WRINKLES ON TANK EXTERIOR ALONG LINES A AND B WERE MEASURED WITH A $4\frac{3}{8}$ INCH LONG STRAIGHT EDGE AT THE FOLLOWING LOADS:

TORQUE LOAD, IN-LBS	WRINKLE DEPTH IN MILS AT LINE A	WRINKLE DEPTH IN MILS AT LINE B
0	< 3	< 3
−100,000	< 3	< 3
−216,000	6	10
+100,000	< 3	< 3
+216,000	< 3	5

CALC			REVISED	DATE	LOCATION ③
CHECK					
A/R					
APR					THE BOEING COMPANY

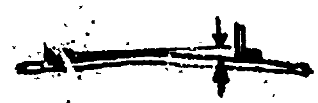
CONTRACT NO.

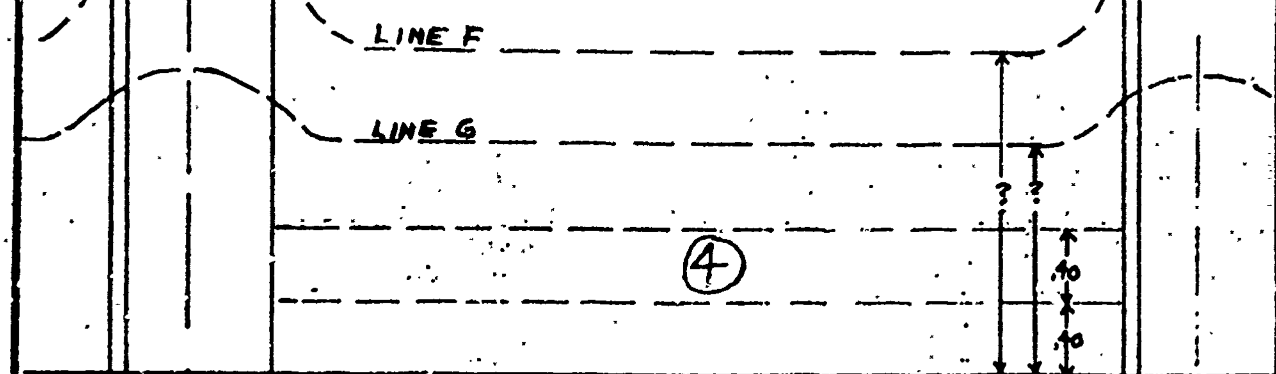
PAGE C-5

DEPTH OF WRINKLES ON TANK EXTERIOR ALONG LINES E, F, AND G WERE MEASURED WITH A $4\frac{3}{8}$ INCH LONG STRAIGHT EDGE AT THE FOLLOWING LOADS:

TORQUE LOAD, IN-LBS	WRINKLE DEPTH AT LINES E	F	G	
0	7	10	8	①
−100,000	5	8	8	①
−216,000	LARGE	12	7	①
℄ LINE E				
+100,000	6	14	6	①
+216,000	LARGE	9	7	(NOW A WRINKLE)

① NOT ACTUALLY A WRINKLE, BUT A CONVEX WEB AS SHOWN:





LOCATION ~~②~~ AND ④

CONTRACT NO.

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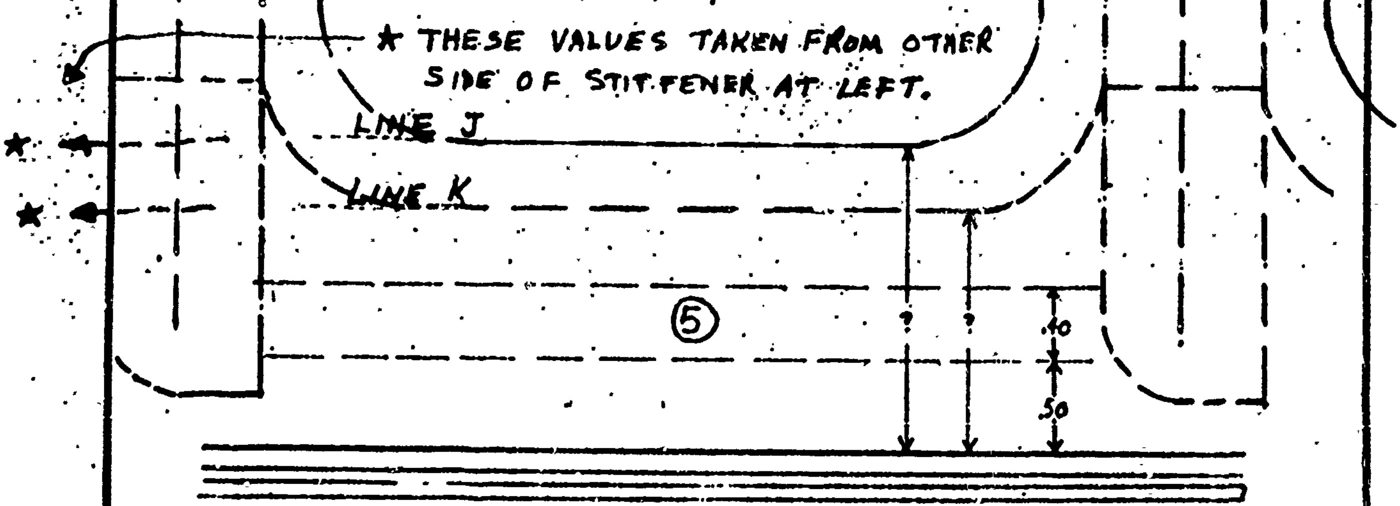
DEPTH OF WRINKLES ON TANK EXTERIOR ALONG LINES H, J, AND K WERE MEASURED WITH A 4 $\frac{1}{2}$ INCH LONG STRAIGHT EDGE AT THE FOLLOWING LOADS:

TORQUE LOAD, IN-LBS	WRINKLE DEPTH IN MILS AT LINE H	J	K	
0	3	3	3	
-100,000	3	<3	<3	
-216,000	L	5	4	
+100,000		~5	<3	①
+216,000	L	13*	9*	①

LINE H

① NOT A WRINKLE, BUT CONVEX WEB AS SHOWN: 

* THESE VALUES TAKEN FROM OTHER SIDE OF STIFFENER AT LEFT.



LOCATIONS ~~4 AND~~ 5 (VIEWED FROM TANK EXTERIOR)

CONTRACT NO.

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REVISIONS			
LTR	DESCRIPTION	DATE	APPROVED
A	To add additional data to the document--covers work conducted from the beginning of the program August 1969 until cancellation March 25, 1971.	4-8-71	L. Middleton R. Billing
B	To add additional data to the document--covers work conducted from August 1971 to December 23, 1971.	1-5-72	L. Middleton R. Billing
C	To add work conducted from December 23, 1971 to January 31, 1972.	1-31-72	L. Middleton R. Billing
D	To add work conducted from February 1, 1972 to January 1974.	2-18-74	L. Middleton R. Billing
E	To add work conducted from January 1974 thru March 1975.	4-16-75	L. Middleton R. Billing
F	To add data to Table 6 (inadvertently omitted from Revision E).	4-24-75	L. Middleton R. Billing

CHANGE RECORD PAGE	REV LTR F

SECT	PAGES REVISED	PAGES ADDED	PAGES DELETED	REV LTR
	The document has been completely revised. 1 through 114			A
	1,8,49, 52,54,56, 57,81,82, 83,96,97, 113, 114	3.1 49.1 57.1 83.1 104.1 104.2 104.3 104.4 104.5 104.6		B
	1,3.1,8, 17,53,76, 82, 113 & 114	57.2 104.7 104.8 104.9		C
	1 5 6 82 113 114	57.3 57.4 57.5 57.6 57.7 57.8 57.9 57.10 83.2 83.3 83.4 83.5 83.6 83.7 104.10 104.11 104.12 104.13 104.14 104.15 104.16 104.17		D

SECT	PAGES REVISED	PAGES ADDED	PAGES DELETED	REV LTR
		104.18 104.19 104.20		D
	1 3 5 6 7 8 57.10 83.6 83.7 101 113 114	57.11 75.1 83.8 83.9 83.10 104.21 104.22 104.23 104.24	3.1	E
	1 82 113 114			F

APPENDIX B

LONG-LIFE, EASILY APPLIED FLUOROCARBON SEALANTS
FOR SUPERSONIC AIRCRAFT FUEL TANKS

Final Report

by

R. Gilliland
P. Mallard
R. Schubert
L. Morris

Prepared under Contract No. Y555369-5835N

for

The Boeing Company
Commercial Airplane Group
Seattle, Washington

Products Research & Chemical Corporation

Burbank, California

TABLE OF CONTENTS

I. INTRODUCTION

The historical development of aircraft fuel sealants begins with the change from bladder-type fuel cells to integral wing tanks. The need for an easily applied sealing compound to resist the fuel, flexing and the temperatures encountered was met first by polymeric alkyd resins. These proved to be limited in both stability and application properties. Materials based upon polysulfide polymers initially used dispersions in water which could be made to fuse together by means of "chemical softeners." The high shrinkage of such materials and the development of liquid, 100% solids, mercaptan-terminated, polysulfide polymers offered a new formulating tool. The standard lead peroxide-cured polysulfides, however, still suffered from attack by fuel. With the invention of the dichromate-cured polysulfides by Products Research & Chemical Corporation, and later the manganese dioxide cured materials, a truly fuel-resistant material was available for aircraft sealing. These materials are, in fact, the basis for sealing nearly all subsonic commercial and military aircraft in the world today.

The United States Air Force in the mid-1950's recognized, however, that the development of supersonic aircraft would require new polymer developments to produce materials beyond the temperature capabilities of the polysulfide rubbers. Products Research, at that time, under WADD 58-59, showed that fluorocarbon elastomers as well as liquid fluorosilicones had significantly improved high-temperature fuel resistance. However, the development of the fluorocarbons has been hampered by their poor low-temperature properties, their unavailability in high solids liquid forms, and finally by concern over stress corrosion effects on titanium alloys at high temperatures.

As a result of these difficulties, the commercial development of high-temperature aircraft sealants has taken place with the fluorosilicones. These products are not without their problems, however. Even stabilized trifluoropropyl polysiloxanes are subject to reversion when confined, so that long-time retention of properties, especially required for commercial supersonic craft, is not available.

As a result of these limitations of the fluorosilicones, the Air Force has funded programs in which the fluorocarbons are chemically copolymerized with the fluorosilicones to give products of improved reversion resistance while retaining adequate low-temperature properties. The Boeing Company has extensively tested such materials and finds them significantly better than the unmodified fluorosilicones for their requirements. They still show thermal deterioration, however. Likewise, the introduction of major fluorocarbon segments into any polymer gives concern with stress corrosion of titanium. This has led to use of the fuel-resistant cyanosiloxanes in some specialty applications.

In the past fifteen years, work has continued with efforts to produce more tractable sealants based on fluorocarbon backbones as well as polymers with heterocyclic nitrogen, etc. Wright Patterson Air Force Base has formulated a number of sealants based upon low molecular weight, vulcanizable fluorocarbons. Early materials showed varying degrees of stress corrosion with titanium. With the introduction of the fluorocarbon, ECD-487, a DuPont product, a material was available which was shown by the Boeing Company to have improved low-temperature properties along with retention of high-temperature properties and absence of stress corrosion. When this polymer was made into a sealant along the lines of the Air Force work, high shrinkage and bubbling was encountered.

The purpose of this project was to find means of producing practical sealants from available low-temperature fluorocarbons which would be expected to perform well in the supersonic transport designed by the Boeing Company.

II. SUMMARY OF WORK

All goals for the fluorocarbon sealant and tape were met with the exception of slightly lower volume percent solids in the case of the sealant.

The sealant developed under this program is greatly superior in application properties to all known solvent-based fluorocarbon materials, has a volume shrinkage less than one-half that of the solvent-applied types, is safe to use from a flammability standpoint and is much easier to apply and tool. The discovery of a water miscible, "reverse phase," technology operable with fluorocarbon dispersions offers wide opportunities for room temperature fusions of otherwise difficult nonfusible, cracking latices.

The tape developed under the program, based upon ECD-487, has a much wider range of temperature usefulness than the standard Viton B materials.

Two different approaches for producing a practical fluorocarbon sealant were followed. Phase I had as a goal the formulation of an 80 volume percent solids material based on ECD-487 which had easy application properties and a useful life at 550°F--twice that of current materials. The second approach employed ECD-487 in a soft, self-vulcanizing tape form at high solids.

Work undertaken to improve sealant application properties and increase volume percent solids by means of pigment reduction and introduction of low molecular weight modifiers was of minor value. Solvent dispersion studies, especially through use of Viton latex sealants, give materials which closely approach the target goal of 80% solids by volume and greatly improved application properties. A volume percent solids of 70%, with weight percent solids of 83%, was obtained. Low-temperature properties are intermediate between Viton B and ECD-487. Further research to improve stability of the sealants and adhesion to titanium is desirable.

Pre-extruded sealants were developed with not more than 10% by volume of volatiles. These sealants have excellent low-temperature properties, being prepared from ECD-487, and are stable when stored at moderately low temperatures. Methods have been developed for adhering these sealants to titanium. Adhesion to titanium was retained after considerable dry heat exposure.

A number of materials were investigated to improve the life of fluorocarbon sealants. One material, Vanstay 8050, was found to be effective in improving the life of solvent-dispersed and pre-extruded sealant.

III. CONCLUSIONS AND RECOMMENDATIONS

A quantum improvement in application properties of fluorocarbon-based sealants has been achieved by the discovery of means for producing room temperature fusible fluorocarbon latices. Combined with the availability of better low-temperature polymers, serious consideration should be given to such materials in supersonic aircraft sealing applications. Retention of good elastomeric properties at 500°F or lower for long periods was observed.

The pre-extruded tape formulations based upon ECD-487 show generally good temperature range of performance and have merit for configurations where high shrinkage or reversion in confined situations is of concern--such as faying surfaces.

Further work in the development of low temperature fusing fluorocarbon latices would be merited in the areas of evaluation of other fluorocarbon latex polymers, improved primer developments for both sealants and tapes, and exploration of the effects of pH, stabilizers and curing mechanisms on the long-term, high temperature retention of physical properties.

III. PHASE I - IMPROVEMENT OF APPLICATION PROPERTIES

The major limiting factor in the current study of fluorocarbon sealants is their difficult application which results in slow extrusion rates, high shrinkage and entrapped bubbles. The current Viton ECD-487 sealant has only 63.5% solids by weight, or 43.3% solids by volume. The goal of this phase is to prepare a one- or two-component thermosetting sealant which has the basic heat-, fuel- and low-temperature-resistance of the current Viton ECD-487 formulation, but has a volume percent solids of at least 80% and a room temperature viscosity of no more than 35,000 poise.

A. Reduction of Viscosity of Current Type Materials

1. Pigment Reduction

Viton ECD-487, the primary polymer of interest, is inherently quite strong compared with other commonly used fuel tank elastomers. The use of pigmentation by carbon black could be dispensed with if its elimination would increase solids content. The effect of elimination and increasing carbon black content is shown below.

	2211-22	2211-23	2211-36
Viton ECD-487	100.0	100.0	100.0
Maglite Y	15.0	15.0	15.0
Thermax	-----	15.0	60.0
FS-1265	8.0	8.0	8.0
EDA DIBK Ketimine	5.5	5.5	5.5
MEK	78.0	81.5	94.5
Extrusion rate @ 90 psi (1/8" diameter nozzle)	38 gms/min.	26 gms/min.	38 gms/min.
Solids Content			
By weight	62.3%	63.9%	66.6%
By volume	42.0%	43.6%	46.8%
Tensile Strength, psi/ Elongation, %			
Cured 24 hrs. @ 350°F	240/1500	270/1200	250/380
Aged 7 days @ 500°F	1420/1000	1400/950	950/600
Aged 35 days @ 500°F	900/150	1300/120	1360/20

As a nonreinforcing grade of carbon black, Thermax, a medium thermal type, was used, its variation produced little effect on viscosity. Increasing carbon black loading gave a marginal increase in volume percent solids over the basic formula, 2211-23, but lowered elongation and heat resistance. The elimination of carbon black decreased volume percent solids. Note, however, the great improvement in elongation retention with reduction of carbon black.

2. Solvent-Dispersion Studies

Frequently, with high polymer solutions, it is possible to prepare colloidal dispersions which exhibit lower viscosities than true solutions of the polymer. This is based on the principle that molecular aggregates can present less interaction than molecularly dispersed molecules because of their lower surface area. A good example of this principle is found in the preparation of rubber cements where hydrocarbon solutions of rubber exhibit high tack and "web-like" character. Introduction of small amounts of the nonsolvent ethyl alcohol produce coiling of the rubber chains and a reduction in the viscosity and webbing occurs. This phenomena was first investigated by choosing solvents or solvent blends which give appropriate solubility parameter (δ) and hydrogen bonding index (γ) for solution of Viton ECD-487. Solutions were prepared of 15 grams ECD-287 per 200 cc of solvent and viscosity determined with the Brookfield Viscometer, spindle #3 at 10 rpm. Results are shown below:

Solvents	δ	γ	Viscosity(poise)
MEK	9.30	5.40	3.6
Ethyl acetate	9.10	5.20	3.4
Tetrahydrofuran	9.10	5.50	3.9
90% MEK, 10% toluene	9.25	5.18	4.1
80% MEK, 20% toluene	9.22	4.97	4.0
70% MEK, 30% toluene	9.15	4.83	5.5
90% MEK, 10% methanol	9.80	5.70	5.3
70% MEK, 30% methanol	10.85	6.33	4.2

Solvents	δ	γ	Viscosity (poise)
50% MEK, 50% methanol	11.90	6.95	5.8
80% ethyl acetate, 20% methanol	10.18	6.10	3.8
80% tetrahydrofuran, 20% methanol	10.18	6.10	4.0
40% MEK, 30% methanol, 30% toluene	10.74	5.70	4.5
60% MEK, 20% methanol, 20% toluene	10.28	5.60	4.0
20% MEK, 40% methanol, 40% toluene	11.20	5.48	8.6

Lowest viscosity was obtained with the pure solvents. Blending a nonsolvent with the solvent generally increased viscosity. By choice of the appropriate solubility parameter and hydrogen-bonding coefficients, solutions were obtained with as little as 20% solvent, with 80% nonsolvent, but at a considerable sacrifice in viscosity. Later work with water and amides gave better results.

3. Introduction of Low Molecular Weight Modifiers

The preferred material for modifying ECD-487 to reduce viscosity would be a low molecular weight version of ECD-487. The DuPont Company had sampled Wright-Patterson AFB with such a material, but was unable to provide samples for this project. Other low viscosity Vitons available are grades LM and LD-011. These materials do not have the desirable low-temperature flexibility exhibited by ECD-487 or its low molecular weight version. However, it was felt that small quantities could be incorporated into the sealant without materially affecting low-temperature properties. In addition, DuPont supplied samples of LD-487-LV, a lower Mooney version of ECD-487, and VTX-3518, also lower Mooney, but intermediate in low-temperature properties between ECD-487 and Viton A. The following results were obtained:

	2211-17	2211-44	2211-51	2211-56
ECD-487	50	--	--	--
Viton LM	50	--	--	--
LD-487LV	--	100	--	--
LD-011	--	--	100	--
VTX-3518	--	--	--	100
Maglite D	3	--	--	--
Calcium hydroxide	6	--	--	--
Maglite Y	--	15	15	15

	2211-17	2211-44	2211-51	2211-56
Thermax	20	15	20	15
FS-1265	--	8	8	8
Hydroquinone	1	--	--	--
N,N-Dimethyl dodecylamine	2	--	--	--
MEK	37	70	--	62.5
EDA-DIBK ketimine	--	5.5	--	5.5
Shell H-3	--	--	5	--
Methyl Isobutyl Ketone	--	--	13.6	--
Solids Content, by weight	77.5%	67.0%	91.5%	69.7%
Extrusion Rate @ 90 psi (1/8: diameter nozzle)	6.2 gms/min.	22.4 gms/min.	10.0 gms/min.	17.2 gms/min.
Tensile Strength, psi/ Elongation, %				
Cured 24 hrs. @ 350°F	250/1450	280/850	--	800/1000
Aged 7 days @ 500°F	--	1000/530	brittle	960/600

The LD-487LV and VT-X-3518 gave only marginal improvement in solids content. With LD-011 Sealant Grade Fluoroelastomer, a 91.5% solids sealant was obtained, but the sealant bubbled badly and became brittle after seven days' oven-aging at 500°F. The ECD-487/LM blend was intermediate between the LD-011 and ECD-487 in solids content. A very low state of cure was obtained with the hydroquinone/amine curing system used.

4. Viton Latex Sealants

Viton B is also available in a latex form designated Viton L-31 Aqueous Fluoroelastomer Dispersion. This material is supplied at 65% solids at a viscosity of 150 centipoise. This is an extremely low viscosity compared with solvent dispersions of the same solids content of about three million centipoise. Much higher solids content and lower shrinkage are possible if the material can be thickened to a sealant viscosity. While Viton B does not have the desirable low-temperature properties of ECD-487, this work was undertaken to devise means of preparing a latex sealant. The technology developed could be readily transferred to an ECD-487 type latex when such becomes available.

In preliminary experiments, merely thickening the latex to a paste consistency resulted in a material which cracked on drying. Blends of the L-31 latex with a polycyanosiloxane fluid improved handling properties. The addition of acid acceptors such as magnesium oxide, zinc oxide, litharge and Dyphos destabilized the latex. A 50-50 blend of L-31 latex and Viton LM dissolved at 80% in methyl isobutyl ketone was prepared and cured with Maglite Y and hexamethylene diamine. The cured product was not well knit and showed cracks on bending. Dimethyl formamide was then used as the solvent for Viton LM and this material was easily handled and applied as a sealant and dried at room temperature to a good elastomer. Again, the use of conventional acid acceptors destabilized the system. A surface-treated calcium carbonate, OMYA-BLH, which is neutral in pH, was found to be a satisfactory acid acceptor in the Viton L-31 latex system. The incorporation of Viton LM was eliminated because of its relatively poor heat and corrosion resistance. Modifiers such as fluorosilicone oil or polycyanosiloxane fluid were required to obviate cracking of the film. The following formulations were prepared:

		2211-41	2211-42
Viton L-31 Latex		154.0	154.0
Dimethyl acetamide		15.0	15.0
Polycyanosiloxane fluid		-----	8.0
FS-1265 fluorosilicone oil		8.0	-----
OMYA-BLH	dispersion	20.0	20.0
Distilled water	dispersion	19.6	19.6
Witco 900	dispersion	0.4	0.4

Degassed to paste consistency (vacuum removal of water to solids indicated)

Curing agent - 20% aqueous solution hexamethylene diamine - 3.5 parts

		2211-41	2211-42
Weight % solids		82.4%	83.0%
Volume % solids		70.0%	70.5%
Aging at 500°F:	42 days	flexible	flexible
	56 days	flexible	flexible
	84 days	weak and short	brittle

Films were cured three days at 75°F, one day at 140°F, one day at 200°F and one day at 350°F prior to 500°F exposure. Best heat resistance was obtained with the fluorosilicone modifier.

An attempt was made to produce a formulation containing ECD-487 as an external phase with internal latex. This resulted in coagulation and a stable sealant could not be produced. Emulsions of ECD-487 in water and methyl isobutyl ketone were prepared at 16%, 21% and 30% solids. Attempts to prepare a latex sealant from these emulsions resulted in coagulation. Attempts to produce a latex from the emulsion by selective absorption of the methyl isobutyl ketone were unsuccessful.

The formulations below show the effect of decreasing calcium carbonate content and of substituting alumina for calcium carbonate. Increasing calcium carbonate from 20 to 30 parts caused cracking of the cured film.

		2211-53	2211-69	2211-72
Viton L-31 Latex		154.0	154.0	154.0
Dimethyl acetamide		15.0	15.0	15.0
FS-1265		8.0	8.0	8.0
OMYA-BLH		20.0	10.0	----
Tabular alumina, 325 mesh		----	----	20.0
Distilled water	dispersions	4.8	2.4	9.0
Tamol 731		0.8	0.4	0.8
Witco 900		0.2	0.1	0.2

Degassed to paste consistency (vacuum removal of water to solids indicated)

Curing agent - 20% aqueous solution hexamethylene diamine - 3.0 parts

	2211-53	2211-69	2211-72
Weight % solids	83.8%	83.2%	82.6%
Storage Stability: @ 75°F	30 days	--	--
@ 32°F	10 months	--	--
Aging of cured films:			
Tensile Strength, psi/Elongation, %			
14 days @ 500°F	590/310	560/360	450/470
21 days @ 500°F	410/310	650/300	---
35 days @ 500°F	330/200	290/240	310/410
49 days @ 500°F	390/160	245/220	210/320
70 days @ 500°F	flexible	flexible	flexible

Reduction of calcium carbonate or use of alumina improved elongation after heat exposure, but caused corrosion of titanium to occur more rapidly With alumina filler, a rather thermoplastic material was obtained, and it is apparent that alumina is not a satisfactory acid acceptor in Viton latex.

Samples of experimental low-temperature latexes VT-X-3627 and VT-X-3627B were obtained from DuPont. These polymers were said to be intermediate in low-temperature flexibility between ECD-487 and Viton B. The VT-X-3627B was a minor modification designed for potentially improved stability. These latexes were compounded into sealants in a formulation similar to 2211-53 (shown above) except that M-Pyrol (N-methyl, 2 pyrolidone) was used in place of dimethyl acetamide since subsequent work on L-31 latex indicated that the former imparted improved application and stability properties.

Use of the centrifuge was investigated to concentrate the latex. Excessive localized hardening occurred unless the centrifuge was run at much slower speeds for longer periods. Concentration of the latex by decanting excess water after the latex was allowed to settle for several weeks was a more feasible method. Additional wetting agent was added in this case to compensate for that lost in the decanting process.

Preliminary VT-X-3627 and VT-X-3627B formulations were less stable than the Viton L-31 formulations. The former coagulated in a few days at room temperature. Storage at 0°F also resulted in coagulation. A temperature of approximately 32°F was found to be the optimum for storage.

Alkaline materials generally used as acid acceptors in Viton caused coagulation of the VT-X-3627 and VT-X-3627B as well as the Viton L-31 latex.

A preliminary formula was prepared without concentrating the latex. Aging tests were conducted at 500°F, comparing two levels of curing agent.

2211-75

	VT-X-3627 Latex	164.0
	M-Pyrol	15.0
	FS-1265	8.0
dispersion	OMYA-BLH	20.0
	Distilled Water	4.8
	Tamol 731	0.8
	Witco 900	0.2

Degassed to paste consistency (vacuum removal of water to solids indicated)

Curing Agent - 20% aqueous solution hexamethylene diamine

Weight % solids 82.2%

Aging of Cured Films	Curing Agent	
	three parts	four parts
Tensile Strength, psi/ Elongation, %		
Initial - cured 1 day @ 150°F plus 1 day @ 250°F	65/150	110/270
Initial - cured additional 1 day @ 350°F	90/500	150/400
Aged 7 days @ 500°F	280/520	330/370
" 30 days @ 500°F	260/340	320/260
" 60 days @ 500°F	220/260	260/120

Initial tensile strength and elongation were quite low until cure[illegible] 350°F. The lower tensile strength in the sample cured with three parts curing agent was due to greater porosity. The lower amine concentration did improve aging, as noted by the superior elongation after 60 days' exposure at 500°F.

In order to determine the practicality of sealing aircraft with Viton latex sealants, corner sections were constructed of titanium, primed and fillets of Viton latex extruded into the seams. Initial efforts, utilizing sealants such as 2211-75 above, resulted in the formation of large cracks at the corner as the latex dried. The formula was modified by increasing the content of FS-1265 fluorosilicone oil from eight to twelve parts. With this modification, no cracking occurred. (See Plate No. 1)

In an effort to improve stability of the VT-X-3627 and VT-X-3627B sealants, calcium carbonate was omitted from the latex. A ball milled dispersion of calcium carbonate together with hexamethylene diamine was used as the curing agent. Formulations and test results are shown below:

	2211-130	2211-131	2211-132	2211-133
	Part B	Part B	Part B	Part B
VT-X-3627 Latex	164.0	-----	164.0	-----
VT-X-3627B Latex	-----	164.0	-----	164.0
M-Pyrol	15.0	15.0	15.0	15.0
Witco 975	1.5	1.5	-----	-----
FS-1265	12.0	12.0	12.0	12.0
	Part A	Part A		
OMYA-BLH	20.0	20.0	20.0	20.0
Witco 900	0.2	0.2	0.2	0.2
Tamol 731	0.4	0.4	0.8	0.8
M-Pyrol	2.0	2.0	---	---
Distilled water	4.8	4.8	4.8	4.8
Hexamethylene diamine	0.8	0.8	---	---
			Part A	Part A
20% aqueous solution hexamethylene diamine			4.0	4.0

Degassed Part B to paste consistency (vacuum removal of water to solids indicated)

Weight % solids, Part B	79.3%	77.7%	82.4%	80.5%
Storage Stability @ 32°F	94 days	94 days	72 days	108 days
<u>Aging of Cured Films</u> Tensile Strength, psi/ Elongation, %				
18 days @ 500°F	560/280	430/200	560/330	350/300
25 days @ 500°F	480/230	370/140	350/270	300/230
38 days @ 500°F	460/170	290/100	365/180	220/160
52 days @ 500°F	380/70	250/40	280/120	200/100

In both cases, VT-X-3627 was higher in solids content and aged better than VT-X-3627B at 500°F.

Incorporation of the calcium carbonate into the latex, as in 2211-132 and 2211-133, gave higher solids content and better aging--probably due to better dispersion of the pigment. Storage stability was not noticeably improved by omission of the pigment; however, sealants prepared at lower solids content and lower viscosity without pigment have been found to be more stable than those noted above. The suitability of such low solids sealants for use is questionable because of excessive shrinkage and flow. All VT-X-3627 and VT-X-3627B sealants have shown progressive increase in viscosity even on storage at 32°F, so storage times shown above can only be considered approximations. The desirability of a Viton latex with improved stability as well as good low-temperature properties is evident.

Improved ease of manufacture of the latex sealants was found by decanting the settled latex and adding a degassing agent, Foammaster S. A vinyl stabilizer, Vanstay 8050, was added to one of the formulations shown below in an attempt to improve high-temperature resistance by decreasing the tendency to dehydrofluorination. Aging tests were conducted at 550°F to accelerate the test.

		2211-174	2211-177	2211-179
VT-X-3627B		127.0		
VT-X-3627			124.0	124.0
M-Pyrol		15.0	15.0	15.0
FS-1265		12.0	12.0	12.0
Witco 975		1.5	1.5	1.5
Foammaster S		1 drop	1 drop	1 drop
OMYA-BLH	dispersion	20.0	20.0	20.0
Distilled water	dispersion	4.8	4.8	4.8
Tamol 731	dispersion	0.8	0.8	0.8
Witco 900	dispersion	0.2	0.2	0.2
Vanstay 8050		---	---	2.0

Degassed to paste consistency (vacuum removal of water to solids indicated)

Curing agent - 3 parts 20% aqueous solution hexamethylene diamine

	2211-174	2211-177	2211-179
Weight % solids	83.7%	82.5%	82.8%

	2211-174	2211-177	2211-179
Tensile Strength, psi/ Elongation, %			
Initial - cured 1 day @ 158°F plus 4 hrs. @ 250°F, plus 20 hrs. @ 350°F	70/410	120/600	60/440
7 days @ 550°F	260/300	240/300	290/240
14 days @ 550°F	170/120	150/120	260/100
Weight Loss: 7 days @ 550°F	-28.3%	-27.4%	-29.3%
14 days @ 550°F	-39.2%	-37.8%	-44.3%

No improvement in aging was found with Vanstay 8050. Stability in storage at 32°F was not as good as with formulations mixed earlier in the project. This was probably due to changes occurring in the latex on standing over a period of several months.

Corners were sealed with these sealants initially as well as one hour after mixing with the curing agent. No cracking occurred in any case. Application life is considered to be at least one hour.

Adhesion studies were conducted on titanium, MIL-T-9046, Type 3, Composition C-6 Al/4V. Preliminary tests included titanate, several silane-type primers and MIL-C-23377 epoxy-polyamide primer. These were tested with and without a secondary coating of brush-type solvent-based Viton using 2211-43 cured with hexamethylene diamine. Best results were obtained with a silane-type PRC Primer #6, or with MIL-C-23377 epoxy-polyamide primer with a secondary coating, 2211-43, prior to application of the latex sealant. Curing at 350°F was generally required to obtain adhesion. Exposure to 500°F oven-aging resulted in loss of adhesion. Tests were conducted at 400° and 450°F to determine how long adhesion was retained at these temperatures. Due to the limited quantity of latex sealant available, peel adhesion tests were not conducted. The sealant was cut to the metal and manually pulled to determine whether adhesive or cohesive failure resulted. Cohesive failure was considered to be satisfactory adhesion. The following results were obtained:

	2211-177	2211-179
PRC Primer #6 + 2211-43		
9 days @ 400°F	cohesive	cohesive
12 days @ 400°F	cohesive	adhesive
14 days @ 400°F	cohesive	--
2 days @ 450°F	cohesive	cohesive
5 days @ 450°F	adhesive	adhesive
MIL-C-23377 + 2211-43		
2 days @ 450°F	cohesive	cohesive
5 days @ 450°F	adhesive	adhesive

The formula for 2211-43 is shown below:

	2211-43
ECD-487	100
Maglite Y	15
FS-1265	8
Methyl isobutyl ketone	80
MEK	300
	503

Curing agent - 20% hexamethylene diamine in MEK - 8.0 parts

Two additional primers, Chemlok 607 and a polyimide polymer (Araldite P-13N, diluted one to one with dimethyl formamide) were tested with 2211-177. The secondary primer, developed under the work on tape sealants, was 2211-186.

	2211-186
ECD-487	100
Maglite Y	15
Thermax	15
Durez 12686	3
FS-1265	8
Diak No. 1	2
MEK	288
	431

The following results were obtained:

	Chemlok 607	Araldite P-13N
Cured 24 hrs. @ 158°F + 4 hrs. @ 250°F + 20 hrs. @ 350°F	cohesive	partly cohesive
1 day @ 450°F	cohesive	cohesive
2 days @ 450°F	partly cohesive	cohesive
5 days @ 450°F	partly cohesive	adhesive
14 days @ 450°F	partly cohesive	adhesive

IV. PHASE IIA - IMPROVING THE LIFE OF FLUOROCARBON SEALANTS

A. Elimination of Metallic Impurities

It has been shown that metallic catalysts such as iron and zinc greatly destabilize vinyl resins, producing catastrophic dehalogenation at elevated temperature. Use of procedures which introduce a minimum of iron and incorporation of organic soluble metal chelating agents to remove such impurities is indicated.

B. Antioxidants

The unsaturation resulting from loss of halogen as well as any hydrogen originally present is subject to reaction with oxygen of the air. Phenolic antioxidants and organic and metallic phosphites have been used to reduce this attack.

C. Metallic Soaps

Combinations of calcium, cadmium and barium soaps have been found useful in adding to the stability of vinyl chloride polymers.

D. Acid Scavengers

Organic materials which remove the acids of decomposition can greatly extend the life of halogenated polymers since these acids are autocatalytic in their effect on decomposition rates. Materials such as olefins and epoxies are known to provide stability until chemically exhausted.

E. Free Radical Scavengers

Very effective, free radical scavengers are a series of sulfur-bearing compounds along with substituted phenolics. These materials prevent the peroxide formation and decomposition occurring at unsaturated sites.

F. Investigation of Cure Mechanisms on Stability

Mechanisms for curing "Viton" involve removal of halogen acid by various procedures. This halogen acid, frequently present as a dissociable salt at high temperatures (or the fluoride ion), can produce problems. The effect of different cures and degrees of cure on stability were investigated.

Since it is beyond the scope of this project to evaluate all of the available materials of these types, representative materials from each category were chosen:

Formula	Material	Type
2211-145	Nopchelate OS	Chelating Agent
2211-140	Santovar A	Antioxidant
	Mark X	"
	Dyphos	"
2211-143	Vanstay 4030 (barium-cadmium)	Metallic Soap
2211-144	Vanstay 8050 (calcium-zinc)	" "
2211-141	Epon 1001	Acid Scavenger
2211-139	Tris-beta chloroethyl phosphine	" "
2211-137	Catalin CAO-6	Free Radical Scavenger
2211-138	Stabilite White	" " "
2211-136		None

The basic formula, 2211-23, was used for comparison. The ketimine, which is very slow-curing, was replaced by 1.6 parts 85% solution of hexamethylene diamine. The sealants were thinned to a flowable viscosity with MEK, curing agent added, and flowed onto glass plates at a dry thickness of 15 mils. After curing, weight loss and tensile properties were determined after exposure at 550°F.

Formula	Additive	Days @ 550°F	Weight Loss	Tensile Strength, psi/ Elongation, %
2211-136	None	0		570/1050
		4	-22.7%	610/380
		7	-37.3%	860/230
		10	-52.0%	1240/85
2211-137	Catalin CAO-6 2 phr	0		500/1200
		4	-26.4%	670/500
		7	-39.3%	900/200
		10	-52.0%	1100/30
2211-138	Stabilite White 2 phr	0		600/1200
		4	-23.1%	640/450
		7	-34.4%	770/120
		10	-46.7%	1130/20
2211-139	Tris-beta-chloroethyl phosphine, 2 phr	0		270/950
		4	-20.4%	640/450
		7	-32.2%	770/120
		10	-46.3%	1130/20
2211-140	Santovar A 2 phr	0		560/1070
		4	-24.1%	700/300
		7	-37.1%	770/120
		10	-50.6%	1510/30
2211-141	Epon 1001 2 phr	0		530/820
		4	-31.4%	990/135
		7	-50.3%	1420/20
		10	-63.4%	brittle
2211-142	Dyphos, 8 phr	0		1120/700
		4	-46.8%	420/120
		7	---	brittle
2211-143	Vanstay 4030, 2 phr	0		800/870
		4	-26.0%	830/175
		7	-40.4%	1140/50
		10	-54.1%	brittle
2211-144	Vanstay 8050, 2 phr	0		550/820
		4	-20.0%	670/375
		7	-27.3%	860/240
		10	-36.4%	1240/130
2211-145	Nopchelate OS 1 phr	0		520/840
		4	-25.3%	600/460
		7	-41.7%	760/155
		10	-56.3%	1260/15
2211-146	Mark X, 2 phr	0		540/870
		4	-21.3%	590/355
		7	-36.7%	780/120
		10	-48.8%	1510/20

In most cases, the additives used were ineffective or were deleterious to aging of the Viton sealant. One material, Vanstay 8050, an organic mixture of calcium and zinc compounds supplied by R. T. Vanderbilt Co., showed improved aging. Weight loss after ten days at 550°F was considerably lower than for any of the other materials tested and elongation was greater. Additional aging tests indicated that 2 phr (parts per 100 of rubber) and 1.6 phr hexamethylene diamine were optimum quantities. The low film thickness of 15 mils in this test was conducive to rapid aging. A thicker fillet of sealant would be expected to retain flexibility for a longer period at 550°F.

G. Pre-extruded Sealant

Some of the properties of Viton fluorocarbon lend themselves nicely to the use of pre-extruded forms. This is not true of the liquid polymers which are too weak and uncured to be self-supporting. Such pre-extruded forms are widely used in construction in cases where the movement and design do not cause working out of these necessarily thermoplastic materials. This limitation is not present with Viton, however, since it actually cures to a thermoset condition. Further, the slow curing time of the Viton ECD-487 is a distinct advantage since it may be supplied already catalyzed, in a cold condition, and will slowly vulcanize in place. The high strength and viscosity of the polymer now become an advantage and it is not necessary to provide a fluid, easy-working sealant. While application and tooling methods are different, the criteria that make a suitable extrusion are well-defined. Solids contents of 80% or more become relatively simple.

1. Formulation and Tape Criteria

Information gathered in Part A of Phase I can be directly transferred here, but higher solids materials are inherent in this approach since the viscosity limitations disappear and other criteria become significant.

a. Cone Penetrometer

In place of viscosity, the cone penetrometer requirements are used as a control. These values range in the case of construction sealants from 70 mm to 140 mm, the lower values (higher viscosity) being used for the sealants requiring some resistance to deformation. Since Viton cures, this upper limit is more for application ease than for providing deformation resistance (ASTM D-5, 100 gms - 5 seconds).

b. Extrusion of Samples

Each material and shape requires a design allowance in the die to produce the proper cross-section. A laboratory extruder with the simple slot and circular cross-section dies was used, with notice being taken of the relation of formula characteristics to extruded shape and size. A Brabender laboratory extruder, Model 101, equipped with variable speed roll feeder and oil-heated barrel with Thermotron #1001 Controller, was used. The extrusion process is shown in Plate 2.

2. Goal

An easy-to-use, self-sticking (to suitable primer or brush-type Viton) extrusion with all the fuel and heat resistance of the liquid sealant containing not more than 10 volume percent of volatiles.

Preliminary tape formulations were prepared as shown below:

	2211-181	2211-182	2211-183
ECD-487	100.0	100.0	100.0
Maglite Y	15.0	--	--
Calcium oxide	--	15.0	15.0
Thermax	15.0	15.0	15.0
FS-1265	8.0	8.0	8.0
Vanstay 8050	2.0	2.0	2.0
HMDA	1.6	--	1.6
Diak No. 3	--	4.0	--
MEK	8.0	8.0	8.0

	2211-181	2211-182	2211-183
Extrusion properties	poor	good	poor
NVM of extruded tape, %	96.2	96.5	--
Penetrometer, 1/10 mm	27.0	31.0	--

Formulations cured with HMDA (hexamethylene diamine) extruded poorly, apparently because of incipient cure. Use of a blocked amine such as Diak No. 3 is necessary for good extrusion. Diak No. 3 is N,N'-dicinnamylidine - 1,6 hexane - diamine. Diak No. 1 (hexamethylene diamine carbamate) was next investigated.

	2211-184	2211-185	2211-186	2211-188
ECD-487	100.0	100.0	100.0	100.0
Maglite Y	--	15.0	15.0	15.0
Calcium oxide	15.0	--	--	--
Thermax	15.0	15.0	15.0	15.0
FS-1265	8.0	8.0	8.0	8.0
Vanstay 8050	2.0	2.0	--	2.0
Durez 12686	--	--	3.0	--
Diak No. 1	2.5	2.5	2.0	2.0
MEK	8.0	8.0	8.0	8.0
Extrusion properties	good	good	poor	poor
NVM of extruded tape, %	96.0	95.7	96.7	96.5
Penetrometer, 1/10 mm	31.0	31.0	28.0	29.0

Formulations prepared with 2.5 parts Diak No. 1 extruded well, but with 2.0 parts, extrusion was poor. The plasticizing effect of the higher amine content is beneficial to extrusion. Either calcium oxide or Maglite Y can be used as acid acceptor with 2.5 parts Diak No. 1 to obtain satisfactory extrusion. Durez 12686 was added to 2211-186 in an effort to improve tackiness of the tape for adhesion to metal. A portion of 2211-186 was thinned to brush consistency for use as a secondary primer for adhesion to titanium.

The best results on oven-aging at 550°F were obtained with 2211-181 and 2211-186. However, both of these formulations extruded poorly. Results are shown in the following table.

Aging of Tape Formulations
(Tensile Strength(psi)/Elongation(%))

	2211-181	2211-182	2211-184	2211-185	2211-186	2211-188
Cured:						
24 hrs. @ 158°F	220/1100	105/600	350/900	320/980	130/700	250/900
+ 4 hrs. @ 250°F	300/910	325/1100	570/540	720/620	250/700	470/650
+ 20 hrs. @ 350°F	560/880	630/860	690/500	730/500	360/730	610/590
Aged:						
7 days @ 550°F	700/300	430/230	600/110	650/210	640/320	550/260
14 days @ 550°F	1220/75	brittle	710/20	810/60	990/80	900/60

Weight Loss

Aged:						
7 days @ 550°F	-25.0%	-32.2%	-30.4%	-26.1%	-27.3%	-36.5%
14 days @ 550°F	-43.1%	-53.1%	-49.9%	-44.2%	-44.2%	-52.2%

Experiments were conducted in adhering the tape to titanium. A double primer system was used, the second coat consisting of a brush-type Viton sealant. The tape was washed with MEK and rolled down into the primer when the latter was barely dry as shown in Plate 3. As the tape is inherently not very tacky, it must be applied while a little solvent still remains in tape and primer. The panels were dried four days at room temperature prior to exposure in the 158°F oven. The following results were obtained:

Tape First Primer Second Primer	2211-181 PRC Primer #6 2211-43	2211-181 MIL-C-23377 2211-43	2211-182 PRC Primer #6 2211-43	2211-182 MIL-C-23377 2211-43
24 hrs. @ 158°F				
+ 4 hrs. @ 250°F	adhesive	cohesive	adhesive	cohesive
+ 20 hrs. @ 350°F	adhesive	cohesive	adhesive	cohesive

Tape	2211-181	2211-181	2211-182	2211-182
First Primer	PRC Primer #6	MIL-C-23377	PPC Primer #6	MIL-C-23377
Second Primer	2211-43	2211-43	2211-43	2211-43
1 day @ 450°F	adhesive	cohesive	cohesive	cohesive
6 days @ 450°F	adhesive	cohesive	cohesive	cohesive
8 days @ 450°F	adhesive	adhesive	cohesive	cohesive
14 days @ 450°F	adhesive	adhesive	cohesive	cohesive
19 days @ 450°F	adhesive	adhesive	adhesive	adhesive

The best results were obtained with the Diak No. 3 cured formulations 2211-182. PRC Primer #6 and MIL-C-23377 epoxy-polyamide primer were equal in retention of adhesion at 450°F, but the latter had better initial adhesion. The following formulations were prepared and extruded:

	2211-190	2211-191	2211-192	2211-193
ECD-487	100	90	100.0	100.0
Viton LM	--	10	--	--
Maglite Y	15	--	--	--
Maglite D	--	15	15.0	15.0
Thermax	15	15	15.0	15.0
FS-1265	8	8	8.0	8.0
Vanstay 8050	2	2	2.0	--
Diak No. 1	--	2	--	--
Diak No. 3	4	--	3.2	3.2
Durez 12686	--	--	--	3.0
MEK	8	8	12.0	8.0
Extrusion properties	good	poor	fair	poor
N/M of extruded tapes, %	96.0%	96.2	94.0	96.17
Penetrometer, 1/10 mm	32	32	35	31

The plasticizing effect of the higher amine content is evident in the above results. The formula with four parts Diak No. 3, 2211-190, had the best extrusion properties. Additional MEK (in 2211-192) was not as effective as the higher Diak No. 3 content. The use of low molecular weight Viton LM (in 2211-191) did not improve extrusion. Aging test results are shown on the following page.

	Tensile Strength(psi)/Elongation(%)			
	2211-190	2211-191	2211-192	2211-193
Cured 24 hrs. @ 158°F + 4 hrs. @ 250°F	180/1200	680/400	380/1100	240/1050
Cured 24 hrs. @ 158°F + 20 hrs. @ 350°F	670/820	920/300	640/920	900/700
Aged 7 days @ 550°F	660/300	870/30	500/230	540/250
Aged 14 days @ 550°F	1300/20	Brittle	1000/50	1120/30
	Weight Loss			
Aged 7 days @ 550°F	-28.9%	-39.6%	-22.7%	-24.1%
Aged 14 days @ 550°F	-47.5%	-62.5%	-41.6%	-42.6%

The formulation containing ten parts Viton LM, 2211-191, exhibited poor aging. Best aging results were obtained with formulations containing the lower Diak No. 3 content.

Adhesion studies were conducted using a pretreatment of the titanium surface with Pasa-Jell 107 prior to priming.

	PRC Primer #6 2211-186 primer		Chemlok 607 2211-186 primer		Araldite P-13N diluted 50% w/DMF 2211-186 primer	
	2211-190	2211-192	2211-190	2211-192	2211-190	2211-192
Cured 24 hrs. @ 158°F + 4 hrs. @ 250°F	cohesive	adhesive	cohesive	cohesive	adhesive	adhesive
Cured 24 hrs. @ 158°F + 20 hrs. @ 350°F	cohesive	cohesive	cohesive	cohesive	adhesive	adhesive
1 day @ 450°F	cohesive	cohesive	cohesive	cohesive	cohesive	cohesive
2 days @ 450°F	adhesive	adhesive	adhesive	cohesive	cohesive	cohesive
14 days @ 450°F	--	--	--	cohesive	cohesive	cohesive
21 days @ 450°F	--	--	--	adhesive	cohesive	cohesive
28 days @ 450°F	--	--	--	--	cohesive	cohesive
35 days @ 450°F	--	--	--	--	cohesive	cohesive
63 days @ 450°F	--	--	--	--	cohesive	cohesive

The polyimide resin primer, Araldite P-13N, was superior in retention of adhesion at 450°F. However, it requires a higher temperature to secure initial adhesion, and good initial adhesion was not obtained with this primer system until the panels were exposed to 450°F.

A small quantity of LD-48/-LV, a lower Mooney version of ECD-487, was obtained. This was evaluated in comparison with ECD-487 in a formulation using 3.6 parts Diak No. 3. This represents a compromise between the superior aging with 3.2 parts and the better extrusion properties with 4.0 parts Diak No. 3. In addition, formulations were prepared from each polymer with FS-1265 fluorosilicone oil omitted.

	2211-197	2211-198	2211-199	2211-200
ECD-487	100.0	--	--	100.0
LD-487-LV	--	100.0	100.0	--
Maglite Y	15.0	15.0	15.0	15.0
Thermax	15.0	15.0	15.0	15.0
FS-1265	8.0	8.0	--	--
Vanstay 8050	2.0	2.0	2.0	2.0
Diak No. 3	3.6	3.6	3.6	3.6
MEK	10.0	8.0	10.0	12.0
Extrusion properties	good	good	fair	fair
NVM of extruded tape	95.0%	95.9%	94.8%	93.9%
Penetrometer, 1/10 mm	29	30	30	34
Tensile Strength(psi)/Elongation(%)				
Cured 24 hrs. @ 158°F + 4 hrs. @ 250°F	380/1100	340/1000	460/1100	420/1200
Cured 24 hrs. @ 158°F + 20 hrs. @ 350°F	710/860	600/840	680/1050	650/1100
Aged 7 days @ 550°F	550/370	690/270	680/230	430/250
Aged 14 days @ 550°F	1090/40	1600/20	[illegible]50/15	620/35

Weight Loss

	2211-197	2211-198	2211-199	2211-200
Aged 7 days @ 550°F	-20.6%	-25.5%	-24.6%	-22.8%
Aged 14 days @ 550°F	-40.8%	-43.4%	-45.4%	-42.2%

Solids content of the LD-487-LV tape sealant was only slightly higher than that prepared from ECD-487, and there was no noticeable difference in application properties. The heat resistance properties of the LD-487-LV were not as good as those of ECD-487, as noted by the higher weight loss and lower elongation of the former. The omission of fluorosilicone oil was harmful to extrusion properties and made no appreciable difference in heat resistance.

Adhesion studies were conducted with and without the Pasa-Jell 107 treatment of titanium, using Araldite P-13N diluted 50% with DMF and comparing secondary primers with and without FS-1265 fluorosilicone oil. A portion of 2211-200 was thinned to brush consistency for use as a secondary primer. Test results are shown below:

Araldite P-13N
2211-186 Primer
No Pasa-Jell 107

	2211-197	2211-198	2211-199	2211-200
Cured 24 hrs. @ 158°F + 4 hrs. @ 250°F + 20 hrs. @ 350°F	adhesive	adhesive	adhesive	adhesive
1 day @ 450°F	cohesive	cohesive	cohesive	cohesive
6 days @ 450°F	cohesive	cohesive	adhesive	adhesive
8 days @ 450°F	adhesive	adhesive	--	--

Araldite P-13N
2211-200 Primer
No Pasa-Jell 107

	2211-197	2211-198	2211-199	2211-200
Cured 24 hrs. @ 158°F + 4 hrs. @ 250°F + 20 hrs. @ 350°F	adhesive	adhesive	adhesive	adhesive

	2211-197	2211-198	2211-199	2211-200
1 day @ 450°F	cohesive	cohesive	adhesive	cohesive
2 days @ 450°F	cohesive	cohesive	50% coh.	cohesive
6 days @ 450°F	cohesive	cohesive	adhesive	adhesive
8 days @ 450°F	cohesive	adhesive	--	--
10 days @ 450°F	40% coh.	--	--	--
30 days @ 450°F	40% coh.	--	--	--

Araldite P-13N
2211-186 Primer
Pasa-Jell 107

	2211-197	2211-198	2211-199	2211-200
Cured 24 hrs. @ 158°F + 4 hrs. @ 250°F + 20 hrs. @ 350°F	adhesive	adhesive	adhesive	adhesive
1 day @ 450°F	cohesive	cohesive	cohesive	cohesive
6 days @ 450°F	cohesive	cohesive	cohesive	cohesive
8 days @ 450°F	cohesive	cohesive	cohesive	adhesive
10 days @ 450°F	cohesive	cohesive	30% coh.	--
21 days @ 450°F	cohesive	cohesive	30% coh.	--
23 days @ 450°F	cohesive	cohesive	adhesive	--
30 days @ 450°F	cohesive	cohesive	--	--

Araldite P-13N
2211-200 Primer
Pasa-Jell 107

	2211-197	2211-198	2211-199	2211-200
Cured 24 hrs. @ 158°F + 4 hrs. @ 250°F + 20 hrs. @ 350°F	adhesive	adhesive	adhesive	adhesive
1 day @ 450°F	cohesive	cohesive	cohesive	cohesive
6 days @ 450°F	cohesive	cohesive	adhesive	adhesive
8 days @ 450°F	cohesive	cohesive	--	--
10 days @ 450°F	cohesive	30% coh.	--	--
14 days @ 450°F	cohesive	adhesive	--	--
17 days @ 450°F	50% coh.	--	--	--
23 days @ 450°F	50% coh.	--	--	--
27 days @ 450°F	adhesive	--	--	--

Test results above confirm the improvement in adhesion with Pasa-Jell 107 treatment and Araldite P-13N plus 2211-186 Primer. This secondary primer, containing FS-1265 Fluorosilicone Oil, was superior to 2211-200 Primer which contained no FS-1265.

The tape sealants, 2211-197 and 2211-198, which contained FS-1265, also retained adhesion for a longer period at 450°F than 2211-199 and 2211-200, which contained no FS-1265.

V. RAW MATERIALS

Viton ECD-487	E. I. DuPont de Nemours & Co.
Viton LD-487-LV	"
Viton LM	"
Viton LD-011	"
Viton VT-X-3518	"
Viton VT-X-3627 Latex	"
Viton VT-X-3627B Latex	"
Viton L-31 Latex	"
Diak No. 1	"
Diak No. 3	"
Maglite D	Merck & Co.
Maglite Y	Merck & Co.
Thermax	R. T. Vanderbilt Co.
FS-1265, fluorosilicone oil	Dow Corning
EDA-DIBK Ketimine (ethylene diamine diisobutyl ketone)	Synthesized at Products Research & Chemical Corporation
MEK (methyl ethyl ketone)	Shell Chemical Co.
MIK (methyl isobutyl ketone)	" " "
Calcium hydroxide	Baker Chemical Co.
Calcium oxide	" " "
Hydroquinone	Van Waters & Rogers
N,N-dimethyl dodecylamine	Union Carbide
Shell H-3	Shell Chemical Co.
Dimethyl acetamide	Van Waters & Rogers

Polycyanosiloxane	Products Research & Chemical Corporation
OMYA-BLH	Pleuss-Stauffer
Witco 900	Witco Chemical
Witco 975	" "
Tamol 731	Rohm and Haas
Tabular alumina, 325 mesh	Alcoa
M-Pyrol (N-methyl, 2-pyrolidone)	GAF Corp.
Foammaster S	Nopco Chemical Division, Diamond Shamrock
Vanstay 4030	R. T. Vanderbilt Co.
Vanstay 8050	" "
Durez 12686	Hooker Chemical Corp.
Nopchelate OS	Nopco Chemical Division, Diamond Shamrock
Santovar A	Monsanto Chemical Co.
Mark X	Argus Chemical Co.
Dyphos	National Lead Co.
Epon 1001	Shell Chemical Co.
Tris-betachloroethyl phosphine	Monsanto Chemical Co.
Catalin CAO-6	Catalin Corp. of America
Stabilite White	R. T. Vanderbilt Co.
HMDA (hexamethylene diamine)	E. I. DuPont de Nemours & Co.

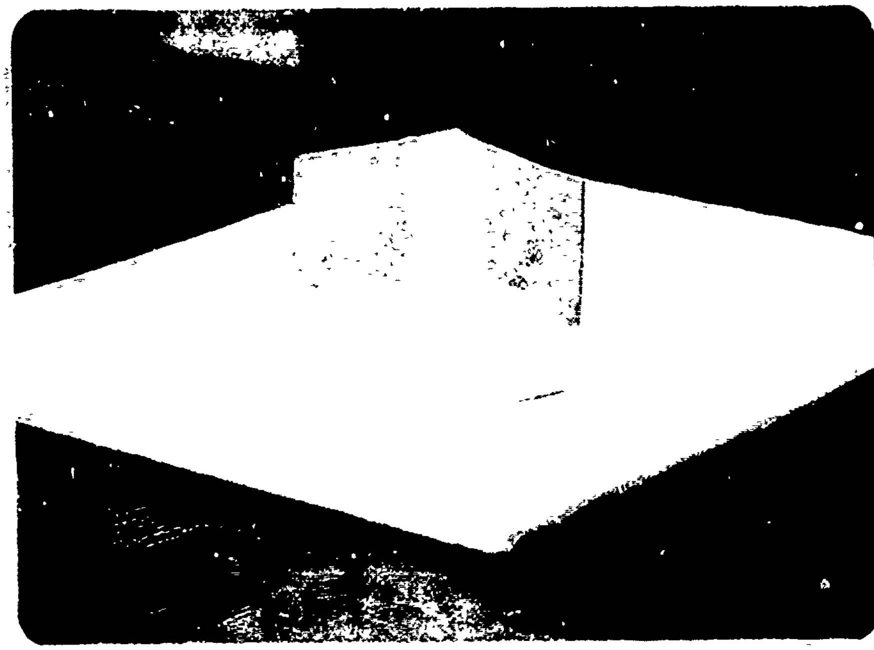
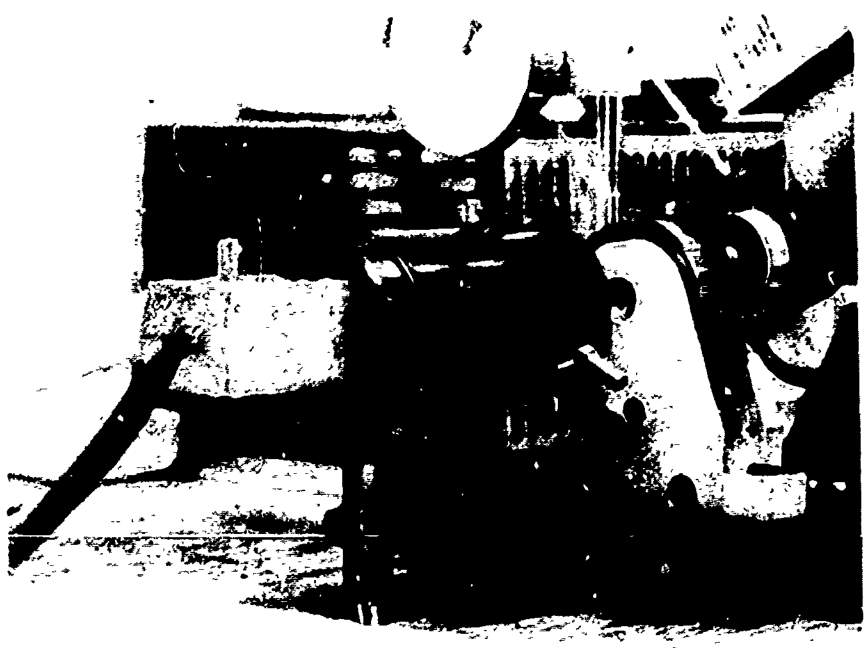


Plate No. 1



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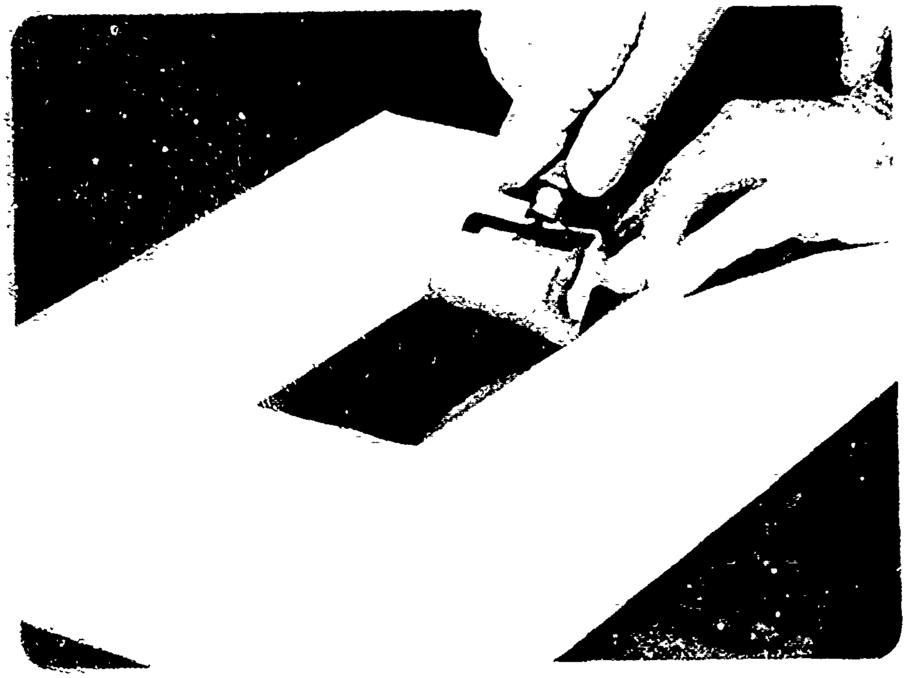

Plate No. 3